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A quarterly paper devoted to the sugar interests of Hawaii and issued by the Experiment Station for circulation among the plantations of the Hawaiian Sugar Planters' Association.

In This Issue:

Soil and Plant Material Analyses by Rapid Chemical Methods:

In the fall of 1932 the manager of a sugar plantation on the Island of Kauai became interested in the potential usefulness of certain popular soil testing "kits." His staff conducted an experimental trial and study of two of these kits, finding, in due course, quite satisfactory correlation of "kit" analytical data with corresponding figures secured by more elaborate analytical procedure.

Previous to this the Director of the Agricultural Extension Division of the University of Hawaii had made experimental tests with a number of kits and in these studies he had found them reasonably reliable, and capable, he thought, of improvement and development for more exacting usage.

The ease and rapidity with which "kit" analyses could be made gave rise to the suggestion that plantation workers generally might employ them in making field nutrient soil surveys and adapt them for other analytical purposes in the various problems confronting the plantation staffs.

The popularity of the kits grew rapidly. Other plantation men added them to their experimental equipment. In the summer of 1933 the Experiment Station, H.S.P.A. in Honolulu began a critical study of kit procedures in an effort to correct certain inadequacies of technic which were found to apply more particularly to the more common types of Hawaiian soils.

This article depicts in some detail the circumstances relating to the early pioneering studies of soil testing kits on the plantations and the attempt to modify kit procedures to make them applicable to Hawaiian conditions.

A sketch is presented of the plantation and Experiment Station cooperative plan of soil nutrient survey in the early 1920's. Circumstances are cited which led to the abandonment of the citric soluble survey and to the adoption of rapid chemical methods of soil analysis (in supplanting the citric soluble process).

Descriptions appear in the article bearing upon the researches conducted in the development of new and rapid methods of soil and plant analyses.

Comments are made upon the growth of plantation interest in making soil and plant nutrient studies upon the plantation premises. The development of separate agricultural-chemical laboratories on plantations is discussed together with the co-

operative activities of the Experiment Station in Honolulu in contributing to the progress of this work.

The transition from kit analyses to the more reliable rapid chemical methods (R.C.M.) is described. The coordination of activities in agricultural-chemical studies by workers in the Industry is presented in this paper as the fabric upon which the R.C.M. enterprise has come into being.

Sections of the paper are devoted separately to descriptions and discussions, respectively, of each rapid method of analysis employed and to detailed procedures followed in conducting all R.C.M. studies.

Formulae and instructions are included for preparing and standardizing the reagents employed in the work.

In August 1936 Hawaiian plantation and Experiment Station R.C.M. studies embraced an organization consisting of forty-three individual laboratories and about one hundred fifty analysts, agriculturists, chemists and other workers.

Soil and Plant Material Analyses by Rapid Chemical Methods

By Francis E. Hance

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Foreword

The one per cent citric acid soluble method of soil analysis has been supplanted almost entirely on Hawaiian sugar plantations by rapid chemical methods* of soil and

* Popularly referred to in Hawaii and hereafter in this paper as R. C. M.

plant analyses. With few exceptions these analytical determinations are being made by plantation men in plantation agricultural-chemical laboratories. A cooperative enterprise consisting of plantation and Experiment Station workers has come into being, having received its initial impetus as the result of the pioneering efforts of a few plantation men and members of the Extension Service Staff of the University of Hawaii.

It is the purpose of this article to discuss the development of the present-day R. C. M., a portion of which had its foundation in the earlier "kit" ensembles. However, before going into details of this development, a better understanding may be realized of the objectives sought in R. C. M. if a brief review is presented of the general plan of the one per cent citric acid soluble soil analysis project and if a few of the more prominent reasons which led to its decline are cited.

The Citric Acid Soluble Project

For purposes of discussion the "citric soluble" project may be divided into:

1. Collection of soil samples.
2. The plantation survey.
3. The analyses.
4. The report to the plantation.
5. Interpretation of data.
6. Reasons for its decline.

1. Collection of Soil Samples:

Two members of the Experiment Station Chemistry Department would visit a plantation, sampling, usually, all the fields upon which cane was growing or which, at that time, were in temporary fallow or in preparation for harvesting, planting, or ratooning. Three plantation laborers would be assigned to assist the visiting chemists. Soil borings were collected, as a rule, from 12 points about 75 feet apart in a field or part of a field. These specimens would be taken from the first foot, and occasionally from the second- or third-foot zones. The 12 borings would then be composited into one sample, about 15 pounds being retained for analysis.

2. The Plantation Survey:

For purposes of expediency and economy the visiting Station men would endeavor to obtain representative soil samples from every field under cultivation, provided it were possible to penetrate growing fields and get about in them. Depending upon the size of the plantation and upon the ground to be covered, the survey would require 1, 2, 3 days, or longer. Usually other plantations on the island would be surveyed during the trip.

3. The Analyses:

Upon arrival in Honolulu the soil specimens would be air-dried, disintegrated by pounding, screened and placed in labeled glass containers. Total phosphates and potash were determined as a rule and also potash, phosphate and lime extractable by strong hydrochloric acid. Chemical treatment of the soil for the one per cent citric

acid determinations consisted of a primary overnight maceration of soil and citric acid solution, adjustment being made the following day to a trifle over one per cent free acid in the soil-solution mixture. The extraction was concluded with a continuous mechanical agitation of the mixture for 6 hours. The extracted soil nutrients determined in the analysis were: potash, phosphate, lime, and silica.

Soil reaction (pH) determinations were made immediately following the receipt of the collected specimens in the laboratory.

4. The Report to the Plantation:

The time elapsing between the collection of soils on the plantations and the mailing of the data and recommendations from Honolulu usually varied between 3 months and 1 year. The duration of this interval was governed by the amount of similar work already in process or being held for attention in its turn and by the number of chemists available to carry on.

5. Interpretation of Citric Soluble and Associated Data:

The value of "total" and "strong hydrochloric acid soluble" nutrient data cannot be questioned, for they serve a useful purpose. Except in a relative sense, however, total and hydrochloric acid soluble figures have little value in arriving at the amounts of soil nutrients present in an immediately available form. The one per cent citric acid soluble data were considered as representing "available" soil constituents. It has been the custom to recommend specific fertilization when the citric soluble potash figure fell below .04 per cent and the phosphate below .005 per cent.

The employment of "citric soluble" data as a measure of availability has never received the unanimous support of plantation men nor of the majority of research workers in the field of soil science. It is not the purpose of this paper to include discussion bearing upon objections to the citric soluble method of soil nutrient appraisal except insofar as the objections apply to the abandonment of the method in favor of the more rapid analyses which are now in vogue.

6. Reasons for Its Decline:

With the foregoing brief summary, chiefly of citric soluble technic, we arrive at a consideration of the reasons given by a number of plantation men for not feeling entirely justified in attempting to use citric soluble data in the formulation of a *current* program of field fertilization.

The nature of the survey renders it expedient to include, for the purpose of soil collection, every field on the plantation that it is possible to enter. This situation entails the sampling of areas at various intervals following fertilization and at various stages of cane growth. Instances have been noted in which fields of young cane have been sampled a week or even a few days following the application of mixed fertilizer. Another objection, equally as serious, has been the unavoidable but quite unsatisfactory delay which necessarily ensues before the analytical data are made available to the manager and his staff. A third objection frequently voiced has been inadequate soil sampling, not only regarding the questionable matter of any given sample representing the immediate fertility of the location from which it may

have been collected—we still have that problem—but more strongly against a composite soil specimen selected from a single random field block which is not equal, in some cases, to one twenty-fifth, or at times to one one-hundredth or less of the area it is expected to represent. Here again we find a fault not due to any inherent defect of the citric soluble survey, except wherein limitations of Experiment Station personnel, costs of analyzing the soils in Honolulu, equipment and chemists to handle a great number of soil specimens (comparable to those collected for the plantation rapid analyses) render a more detailed general survey an economic impossibility. Individual 2-acre, or even 5-acre soil composites collected from harvested fields of all plantations could scarcely be analyzed regularly in Honolulu by the old methods with a chemistry staff of approximately 15 analysts. But it should be noted that this accomplishment has been achieved with R. C. M. on several plantations within a harvesting season by one trained analyst and a helper.

The Introduction of Soil-Testing Kits in Hawaii

The foregoing discussion is offered as a background to clarify somewhat the dissatisfaction of plantation men which made it evident that a radical change in the method of soil analysis was not only imminent, but urgently needed.

Rapid Chemical Analyses Not a New Idea

The examination of soils by rapid methods of analysis for determining concentrations of available plant nutrients is not a recent innovation. In his text on *Soils* published in 1906, E. W. Hilgard (6) includes an appendix by Loughridge, entitled "Short Approximate Methods of Soil Examination Used at the California Experiment Station." In the text of the appendix Loughridge describes procedures for estimating concentrations of phosphates, lime, humus, and alkali salts in soils, any one of which may be completed within one hour. The determination of potash in soil is also described but the analysis required about 3 days for completion.

Prof. F. G. Krauss and the Extension Service of the University of Hawaii

In his early association with Prof. Hilgard in California, Prof. Krauss, recently retired Director of Agricultural Extension Service, University of Hawaii, found that useful and practical applications of the rapid methods of soil analysis were being made at the California Experiment Station and that in general they had been found satisfactory in classifying agricultural lands and useful in formulating plans for fertilization. Later, in his agricultural extension activities in Hawaii, Prof. Krauss felt the need of rapid analytical methods for examining soils and in due course conducted experimental studies using several Mainland-manufactured, soil-testing kits. He found limitations in the use of the kits and noted features concerning the technic of the kit determinations which were objectionable. Nevertheless, he definitely established the fact that a marked deficiency of any one of several major plant nutrients could be ascertained by kit soil tests. In several cases his kit findings were verified by pot and field plot tests. "Trends" of nutrient deficiencies, as determined by quick soil tests, were substantiated to a degree by chemical analyses conducted by several colleagues.

Grove Farm Investigates Soil Testing Kits

In the autumn of 1931 Prof. Krauss, in a conversation at his office in the University with E. H. W. Broadbent, Manager, Grove Farm Company, Ltd., Kauai, discussed the results he had secured in the studies then under way with the soil-testing kits. He suggested to Mr. Broadbent that the advantages of the rapidly conducted kit tests embraced not only small costs of analysis but that the scheme rendered it possible to examine a large number of individual soil specimens from comparatively small areas. Later, by arrangement with Prof. Krauss, Mr. Broadbent was given, at Lihue, a demonstration of the Urbana kit test (1) for soil phosphate by J. C. Thompson, County Agricultural Extension Agent, University of Hawaii. Following this demonstration, Mr. Broadbent ordered one of the phosphate soil-testing kits and one Urbana potash kit which had just been placed on the market, delivery being made at Lihue in December of 1931. W. P. Alexander, Assistant Manager, Grove Farm Company, Ltd., thereafter conducted, we believe, the first experimental tests of these kits in Hawaii upon sugar cane field soils. In January of 1932 Mr. Alexander established definite correlation between the one per cent citric soluble soil data for potash and phosphate and the groupings of nutrient concentrations found in his studies by the kit tests. He found a marked similarity in trends in comparing the two methods of analysis when the results of either were tabulated by grouping the data in designations of low, doubtful, medium and high.

The Experiment Station Investigates Kit Procedures in the Field

Mr. Alexander continued his studies during 1932 and in June of that year turned over the general investigational details to A. Ayres, assistant chemist, Experiment Station, H.S.P.A., the latter having gone to Kauai to devote his attention to this work.

Mr. Ayres established a laboratory in a building which was very generously placed at the disposal of the Experiment Station by the Lihue Plantation Company, Ltd. He received the cooperation and assistance of A. M. McKeever, B. B. Henderson and other staff members of the plantation in equipping the laboratory. Mr. Ayres proceeded to study field applications of kit analyses, the object being to determine the limitations of the kits and the possibilities of their employment in soil nutrient survey studies. The laboratory was intended originally as headquarters for conducting field studies.

The First Departure From Conventional Kit Technic

In a short space of time Mr. Ayres found it difficult to perform consistently, with any degree of accuracy, certain portions of the prescribed kit soil analyses without introducing numerous modifications which were suggested, of course, by his previous training and experience. His tests ceased to be confined to the equipment of the "laboratory in a tin box." He added several standard laboratory devices to the technic of work and noted hindrances which developed in the prosecution of the tests. He found also that phosphate could not be determined by the kit method on certain Kauai soils, due to the highly colored nature of the extracting solution following contact with the soil. He experienced difficulty in making the potash tests

because of the unsatisfactory method of manually controlled rotary mixing of soil extracts and reagents and because no constant source of artificial light was provided, calibrated in an optical device, to permit reasonably accurate turbidimetric readings in the final potash test solution. These and other improvements were to appear later by other workers but Mr. Ayres was forced to improvise an optical instrument for making test readings in the soil phosphate determination and to filter off the soil extract from the soil specimen preparatory to developing the critical color reaction of the test. Difficulty of a chemical nature developed when he attempted to make phosphate tests on calcareous soils. The extracting solution furnished with the kit is mildly acidified with hydrochloric acid. In the presence of calcium carbonate of the coral this free acidity may be partially or entirely neutralized, the result being a faulty extraction and failure to obtain in solution the available phosphate contained in the soil specimen. This difficulty was also overcome later at the Experiment Station in Honolulu.

Experimental Results of Field Trials Using Slightly Modified Kits

Having determined the limitations of the kit tests and having introduced modifications in the technic of the soil phosphate determination, Mr. Ayres proceeded with the field kit study on Kauai soils with a degree of progress quite satisfactory under the limitations of the study. He found, for instance, that it was a feasible undertaking to sample and analyze a very large number of soil specimens from a single field and to seek in the resulting data information relative to the uniformity of the field with respect to the nutrient in question. In this manner he established the fact that a wide degree of variation existed in the concentrations of available phosphate in the average cane field of Kauai. In cooperation with J. N. P. Webster, then Island Representative of the Experiment Station on Kauai, Mr. Ayres worked up correlations between field experiment and kit data on available soil phosphate. As a result of this work the conclusion was reached by these men that the response of sugar cane to phosphate fertilization on Kauai soils should not be expected if the supply of this nutrient in the field exceeded an amount corresponding to that determined as "doubtful" on the kit scale.

At this time Mr. Ayres was also able to show by the comparison of kit analyses of field soils with adjacent virgin areas that in the case of acid soils practically all of the phosphate extracted in the kit analysis came from added fertilizer and that virgin acid soils, even though containing some available phosphate, failed utterly to give indications of the fact by regulation kit analyses. This finding indicates that naturally occurring phosphates in certain acid soil types are totally insoluble in the kit extracting medium.

A Laboratory Study of the Original Kits in Honolulu

Somewhat prior to Mr. Ayres' work on Kauai, L. E. Davis gave attention to the study of a number of popular soil-testing kits in the chemistry laboratory of the Experiment Station in Honolulu. A final step in the soil phosphate determination of one widely used kit involved the stirring with a tin rod of a supernatent liquid in a test vial above a quiescent settling of the soil specimen under examination. This

operation developed a blue coloration in the test liquid directly proportional in intensity to the amount of phosphate extracted from the soil below it.

Mr. Davis found that, with many Hawaiian soils, this supernatent liquid layer, lying above the soil in the vial, was frequently highly colored with organic matter, iron compounds, etc., and quite often was extremely turbid because of the presence of a finely divided suspension of soil which would not settle for hours or in some instances for days. In cases such as these it was futile to attempt an estimation of the blue color in the orange colored turbid or muddy liquid medium for it is upon the intensity of the blue color developed in this medium that the analyst estimates his quantitative reading. The blue coloration produced with the tin rod is due to the reaction of the tin with compounds of molybdenum and phosphorus. Under properly standardized conditions the intensity of the color thus developed becomes a very accurate measure of the quantity of phosphate present. One of these conditions requires a narrowly defined range of acidity. If the acidity becomes too low, deep blue compounds of molybdenum are formed which contain no phosphate whatever. Obviously, in this circumstance, the color of the solution is of no value in estimating the phosphate extracted from the soil. On the other hand, if the solution is too acid the blue color is not formed even when large amounts of phosphate are present.

Hence, Mr. Davis encountered difficulty as did Mr. Ayres in working with alkaline or coral soils which partially or entirely neutralized the feeble acidity of the kit reagent used in the extraction of the soil. With these difficulties in mind, Mr. Davis attempted to adapt the kits to as wide a use as possible. He too found it essential to depart from the "laboratory in a tin box." Improvement came immediately in the modification of the procedure whereby the extracted soil was removed from the extracting reagent by filtration and where the critical blue color was developed by using a solution of stannous (tin) chloride in place of the tin rod. The test was still restricted, however, to non-calcareous soils. A second improvement, devised by Mr. Davis, with the cooperation of E. K. Hamamura, consisted of an extraction of the soil with dilute hydrochloric acid in place of the kit reagent. The filtered extract was then evaporated almost to dryness and the residue was dissolved in the proper reagent. This change made the method applicable to calcareous as well as non-calcareous soils.

Difficulties With the Potash Soil Kit

In his study of a commercial kit method for the estimation of available potash in soil, Mr. Davis found that the composition of the potash compound producing the turbidity in the test—the important last step in the determination—varied considerably, not only with the amount of potash extracted from the soil, but, unfortunately, with the manner of adding reagents to the soil extract and in the character of the manipulations used by the analyst in the analysis. In this test the estimation of potash involves the formation of sodium-dipotassium-cobaltinitrite in the test solution. This compound is produced by the analytical procedure, generally, in very finely divided form so that the mixture becomes turbid. When properly conducted the turbidity so produced is related directly to the amount of potash

extracted from the soil. The difficulties encountered with the potash kit were overcome later and will be described in more detail.

Extended Experimental Trial of the Kits On a Few Plantations

Interest in the kit methods of soil analysis, while not widespread among Hawaiian sugar plantations at this time (1932), was nevertheless on the increase. But, while interest grew in the kit experiment, dissatisfaction also developed because of the irrational results obtained on our many prevailing soil types and because of the failure of those investigating the methods to secure consistent analytical checks.

It must be emphasized that the principal commercial soil-testing kits were extremely simplified assemblies of apparatus and chemical reagents intended primarily for the individual farmer who may have had little or no experience whatever in using equipment of the kind. Much less would he be likely to devote enough of his time and attention to the study in order to interpret his data in a manner which would include correlations with replicated field experiment and other exacting procedures of scientific soil evaluation.

Some Advantages of Original Kit Assemblies

Before proceeding further in pointing out what we believe to be disadvantages of kit methods in general, it should be emphasized that of all the commercial kits investigated at the Experiment Station in Honolulu, with one exception, it was gratifying to discover that they were not designed to mislead anyone and that they were sound in their fundamental principles. It is also true that had it not been for the simplicity of the kit tests and for the rapidity with which an analysis could be made with them—unreliable as they proved to be with some Hawaiian soils—it is indeed doubtful if the intensive experimental study of rapid chemical methods would have received the stimulus it did among plantation workers.

With one of these kits a farmer could enter his fields, procure a sample of soil and immediately analyze it on the spot. This distinctive "kit" feature led to the undeserved quasi-derisive sobriquet for the kits of "running board laboratories."

Other Objectionable Features of Commercial Kits

The experiences of plantation investigators and the chemistry staff of the Experiment Station soon brought to light the fact that the limitations of the soil-testing kits were many and frequently were serious. They were inaccurate not merely in the sense that results could not be expressed to as high a degree of precision as those obtained from the prevailing analyses of citric acid soil extracts, but in the more important sense that they were in many cases utterly unreliable.

A Plantation Staff Cites Its Kit Experiences

Apropos of the difficulties encountered by plantation investigators we quote in part a letter received by the author in May 1936 from Wm. Campsie, Manager, Hutchinson Sugar Plantation Company, Naalehu, Hawaii. He wrote: "I can remember very well our first experience with those kits. The phosphate kit was good

in certain kinds of soil, but I was tempted many times to scrap the potash kit as we could get no correlations with juice sampling or Mitscherlich tests and could not even duplicate our results on the same soil from day to day.

"Only Hossack's tenacity and perseverance kept us going with it at that time." (The time referred to by Mr. Campsie was in the summer of 1933.)

Mr. Campsie's reference to Mr. Hossack's tenacity and perseverance in staying with the kits is particularly appropriate in its bearing upon the development of R.C.M. as it exists today in Hawaii. One by one, plantation workers were showing unmistakable lack of interest in kit studies. Investigational kit study at the Experiment Station (already referred to) was of necessity slow in bringing about proven modifications which could be recommended for general adoption. Mr. Hossack, however, hung on and in the face of waning Hawaiian interest in the kits his perseverance bridged a gap which otherwise might have brought about the termination of plantation interest in any form of rapid soil analysis.

The author requested Mr. Hossack to recount his experiences in the evolution of the kit tests to the modern rapid chemical methods. He has very kindly contributed an interesting and concise account of his work with Mr. Campsie which is quoted directly below.

The original Urbana soil-testing kits were first used at Hutchinson Plantation about the end of 1932, and by the beginning of 1933 a regular system of soil sampling was inaugurated. A small amount of experimentation was necessary in order to decide on the best method of conducting a soil survey of the whole plantation, but very little time was lost, and an intensive system of soil testing was well under way in January of 1933, and our records of this work are complete from that date up until the present time.

The system that was adopted was to collect a representative number of soil samples from each field immediately after harvest. In the early work 1 composite sample from 5 to 8 acres was the general rule, but as the value of this work was recognized more time was set aside, and with the help of one laboratory assistant more and more samples were collected, so that by the beginning of 1934 it was possible to sample all fields on a 2-acre basis.

A regular system of correlating kit analysis with citric soluble analysis and results from Mitscherlich tests was also started right at the beginning of the soil survey. During the early period it was sometimes difficult to get a correlation between the various methods. Many difficulties were encountered, errors in Mitscherlich tests, faulty reagents and imperfect apparatus, all caused a great deal of inconvenience, but where all factors showed a definite correlation, we were able to conduct a fairly elaborate system of spot fertilization. When there was any doubt we erred on the safe side by applying more fertilizer than was necessary.

There is no doubt whatever but that the gradual perfecting of the kits by the chemistry staff of the Experiment Station did more than anything else to establish confidence in the data secured from the soil survey. Both the phosphate and potash kits were far from being reliable in their original form.

The high organic nature of some of the soils of our upper fields caused a very erroneous estimation of their phosphate content, and it was not until the perfected "Laboratory Method" was introduced that we were able to secure accurate data. Fortunately our lower fields were low in organic matter, and the original kit analysis showed us a fairly accurate picture of our phosphate supply in these areas. In this manner we were able to omit phosphate on a considerable area where all methods of analysis showed a high amount. We did little spot fertilization with potash in the very early days, and it was not until the advent of the Potash Illuminator and later of the Potash Rotator that we were able to place a high degree of confidence in our potash analyses.

The whole history of the development of a systematic soil sampling program on this plantation has been one of continuous improvement. New methods of technic, discarding of

faulty and cumbersome systems, confidence in the data secured, have all helped to establish a system of fertilization control that is saving the plantation thousands of dollars a year and has given us a newer and clearer understanding of our plant food requirements.

The Hawaiian Soil Phosphate Fixation Kit

The former Director of the Experiment Station, H. P. Agee, contributed his support and interest to an exhaustive study by the writer and other chemistry department members of kit technic with Hawaiian soils in an attempt to modify them sufficiently to meet sugar plantation requirements or to develop new or original rapid methods of analysis to meet Hawaiian soil conditions.

One of the first developments in this venture was the appearance of the Experiment Station, H.S.P.A. soil phosphate fixation kit. This rapid methods assembly was developed by the director, the writer and Q. H. Yuen of the Chemistry Department. Its use enabled the cane grower to ascertain the degree of "tie-up" or fixation of phosphate which may be expected in his fields following the application of a soluble phosphate fertilizer. This assembly was later provided with an adjunct which permitted additional estimations to be made of intensity and rate of soil phosphate fixation. This assembly, its development and uses, has been previously described (3). Additional comments will not be made here except, perhaps, to state that the fixation assembly is in wide use today, with no new modifications having been necessary since the addition of the adjunct feature in the fall of 1934.

Developments Regarding Departures From Commercial Kit Procedures

Through a great number of disappointing setbacks, the kit features of the various soil tests gradually gave way to more precise means of estimating the concentrations of available soil nutrients. Procedures were developed, in some cases, where radical modifications were made from the technic of the original kits. New equipment and severe simplicity and straightforwardness of analytical detail were maintained; so likewise was the important feature of rapidity of analysis. As they accrued and were found useful and practical, improvements and newer developments were submitted to the plantation users for criticism and cooperative study. One of the features of the kits first abandoned was the use of the opaque, printed color chart to compare developed test colors with standard printed variations of a definite shade. In its place was substituted clear solutions of organic dyes sealed in vials which were duplicates of the test vials used in the analysis.

Rapid chemical soil analyses would be difficult of accomplishment were the chemist to be deprived of the use of color phenomena as indicators of completed reactions or as measures of concentrations of various soil constituents which lend themselves to quantitative color reactions.

Laboratory analyses in R.C.M. work are, as a rule, conducted in such a manner that the final test solutions are placed in thin glass vials preparatory to the development of the distinct shade of color in clear solution, opacity or turbidity which is directly comparable with the concentration of the nutrient which is sought in the analyses. When "shade of color in clear solution" is the measure of the nutrient sought, comparison of the test with opaque, printed charts (in the ordinary variable sources of light in the laboratory) is far from being a satisfactory or reliable pro-

cedure. For this reason we adopted the long-established system of making color comparisons by the use of a standard source of artificial illumination. The unique features of the departure from kit, printed color charts embraced the selection of vials identical with those used in the various tests for carrying standard colored solutions for comparison purposes, the development of illuminating apparatus, racks in which to make the comparison, etc., and of the standard colored solutions themselves.

The Gradual Evolution From Kit Tests to R.C.M.

With the beginning of studies in the preparation of satisfactory color standards Messrs. Yuen, Hamamura, Nishimura and Chu of the chemistry staff were assigned to various research topics leading to improved technic in analytical procedure. Researches continued as progress was made, these same investigators devoting their entire time to assisting in the development of new chemical methods of analysis and further refinements in established procedures.

Many outstanding chemical and mechanical developments in the present R.C.M. complement of assemblies have been due entirely, originally or individually to the efforts of Messrs. Yuen, Hamamura, Nishimura and Chu.

Dr. H. L. Lyon, Director of this Experiment Station, members of the chemistry and agricultural staffs, the plantation staffs and the author have contributed other details.

To avoid countless repetition and frequent acknowledgment of individual effort in the following description of R.C.M. developments, no attempt will be made to associate in any more detail the growth of the enterprise with any of the personnel who have contributed to the work.

Color Standards

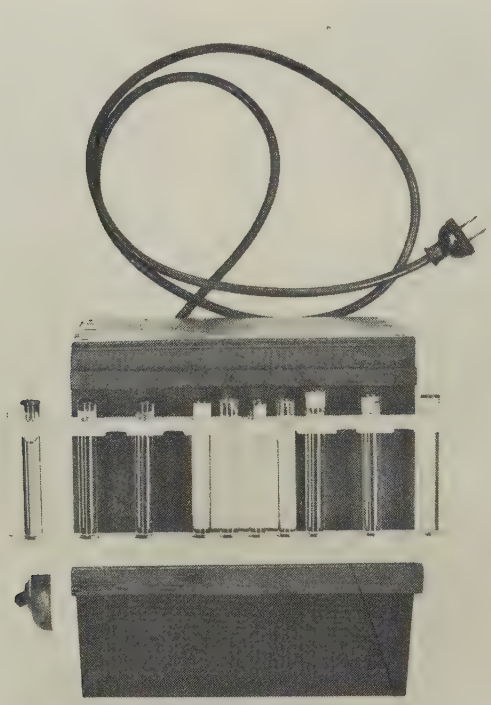
Attempts to prepare durable and permanent liquid color standards were fraught with difficulties. Aniline dye solutions which were fast to light in quartz or "nonsol" glass containers slowly deteriorated when put in service over artificial light in the regulation test vials closed by rubber stoppers. Marked improvements have been obtained in maintaining the permanence of these standards and in extending their useful life by impregnating the container stoppers with a nitro-cellulose compound and in peptising the dye solutions (which are colloidal) with appropriate stabilizers.

Satisfactory standards of an improved nature have been prepared from solutions of inorganic salts wherever the colors obtained from such sources may be used. Research at present is devoted to the substitution of stabilized inorganic permanent standards for the naturally fugitive dye solutions, the latter having been found unable to withstand prolonged usage without gradual deterioration. At the time of this writing (August 1936) the useful life of the most fugitive of the R.C.M. color standards has been extended to four or more months.

Color Comparison and Turbidity Apparatus

The adoption of liquid color standards for comparison purposes in rapid colorimetric analyses necessitated the designing and building of suitable apparatus to conduct the tests. For the determination of phosphate a ventilated wooden box was

designed to enclose a calibrated electric light bulb which could be turned off and on by a switch placed at a convenient point outside. The light within the apparatus was released through a dense, opal glass window built in a plane 45° from the horizontal. By means of a permanently attached recessed guide placed on the outer surface of the device and under the illuminated window, a notched rack can be accommodated. The rack can be made to slide back and forth in front of the window. The sliding rack is provided with circular openings between the notches, the former carrying the color standard tubes in graded shades of increasing intensity from left to right. The notched indentations in the rack were gauged to permit the ready



The H.S.P.A. Phosphate Illuminator.

insertion and removal of the analytical test vial from point-to-point along the rack. By this means the shade of color of the solution under examination can be matched with the appropriate standard in a field of constant, diffused artificial light.

In the potash determination the end point or critical period of the analysis depends upon the measurement of turbidity produced in the test by a finely divided orange-colored precipitate. The Urbana Laboratories (2), the originators of the soil potash kit using this principle, employ a chart in separate gradations of fine, medium and heavily ruled series of lines as a means of gauging the turbidity produced in this test. The vial containing the turbid orange-colored suspension is held by the analyst about one-half inch above the ruled chart and, while sighting down through the test liquid, the vial is moved across the separately spaced groups of ruled lines until any one of the series of lines may be distinguished through the tur-

bid liquid. The ability to just distinguish any one of the lined groups constitutes the final step in the analysis. (More details will appear later under the description of the R.C.M. determination of potash.)

There are two serious objections to this procedure, both as to convenience and consistent accuracy of analyses. Uncertain source of natural light is one objection and difficulty of holding test vials always exactly one-half inch from the chart is another. To overcome these faults the potash illuminator was developed. This device is similar to the box-like phosphate illuminator containing a calibrated electric light bulb and ground glass window. In the potash illuminator the window of the instrument is placed on the horizontal upper surface of the device. In a recessed opening directly above the window is placed a printed lined grid, covered by a piece of clear glass which is just flush with the upper surface of the instrument. A slide, perforated to accommodate 3 test vials, may be moved back and forth in the field of light between 2 lateral guides permanently attached at the edges of the upper surface. By this means turbidity may be measured by sighting through the column of test liquid upon a uniformly lighted field which carries the ruled grid or chart. The 2 illuminating devices just described appeared in the spring of 1934.

The H.S.P.A. Potash Illuminator.

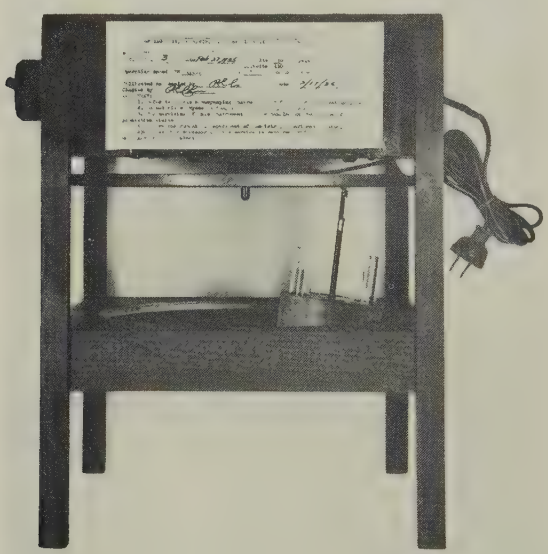
The Potash Rotator

Up to September 1934 some progress had been made in the establishment of individual agricultural soil-testing laboratories among the 39 plantations of the

Association. Progress was slow, however, chiefly because of the difficulties encountered by plantation men in securing consistently accurate checks of analyses of soils for readily soluble potash and phosphate. (Potash, phosphate and soil reaction were the only analyses made at this time.)

The introduction of the Potash Rotator was followed by a marked stimulus of plantation interest in conducting plantation soil studies. This instrument also greatly improved the accuracy and reliability of the determination of potash.

In the early potash kit procedure, the analyst was instructed to agitate 4 drops of a reagent with 1 ml. of soil extract in a short glass vial provided for the purpose. This step was followed by the introduction of 1 ml. of a second reagent so that the latter would form a clear distinct upper layer upon the solutions previously placed in the vial. Up to this point no difficulties were encountered by anyone after having practiced the various steps involved. However, the next operation requires an un-

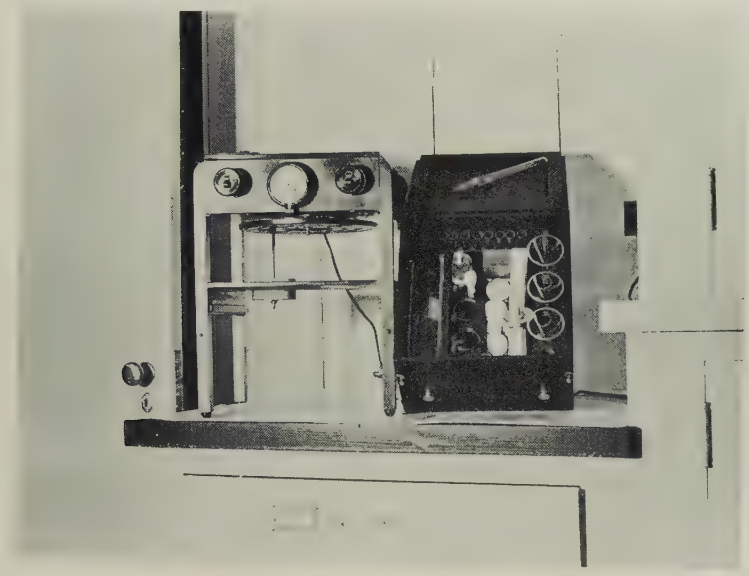


The H.S.P.A. Potash Rotator (side view).

usual degree of skill if consistent results are to be regularly obtained. The analyst takes the vial in his hand and for a period of one-half minute he agitates the contents in a gentle circulatory manner so as to produce a distinct type of crystalline turbidity which is the measure of the potash occurring in his "unknown" soil extract. It was this feature of the potash determination which appeared to be responsible for the widely variable results turned out by even the most experienced analysts doing this work. Our efforts at the Experiment Station in Honolulu were

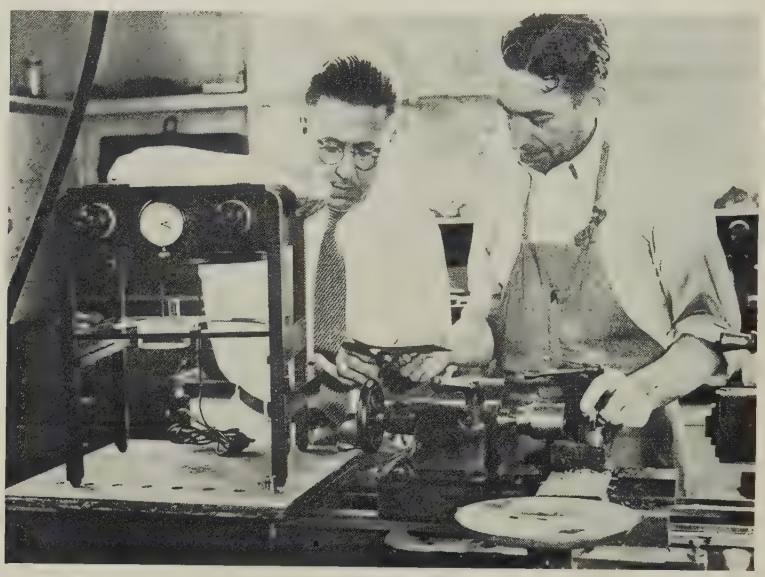
directed towards devising a mechanical instrument which would always perform this difficult operation in the same manner and without variation or change when once correctly adjusted.

In August of 1934 the first Potash Rotator was built which met all the requirements imposed by a very exacting manipulation. Improvements upon the original machine were made from time to time. The present Potash Rotator consists of a

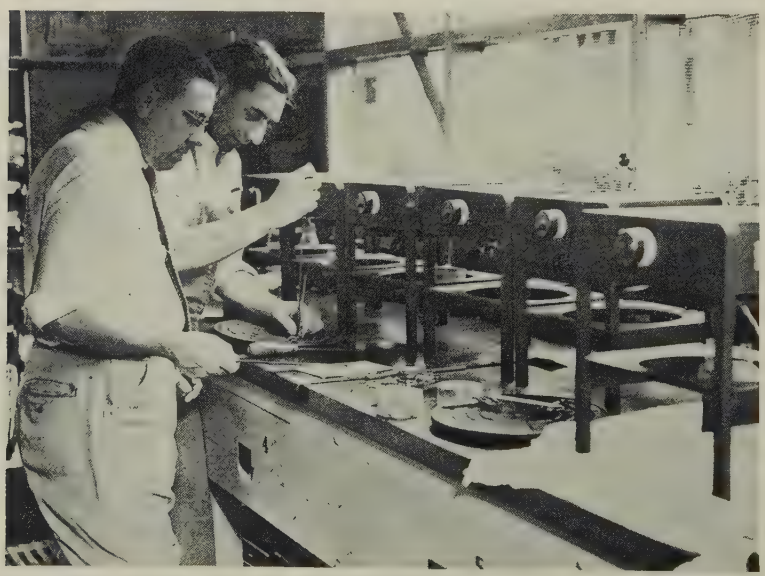


The H.S.P.A. Potash Rotator (portable). Designed for plantation demonstrations.

double-decked frame about 28 cm. square and 36 cm. high. The upper deck supports and encloses an induction-type electrically driven motor and mechanism which has been adjusted to turn a 29 cm.-flywheel in a horizontal plane directly beneath the upper deck. At a distance of 8 cm. from the center of the flywheel, on its lower side, a 3 mm. round brass shaft, 15 cm. in length, is attached by a hinged joint so



The author and W. F. Burton, machinist, in the Experiment Station, H.S.P.A. shop.



H.S.P.A. Potash Rotators being manufactured for shipment to plantation R.C.M. laboratories.

that it hangs, normally, in a vertical position (at right angles to the plane of the flywheel) and tends to be thrown out from the center of the flywheel by centrifugal force when the wheel is turning. The brass shaft supports a disc-like, rubber-covered wooden rotor, the latter 8 cm. in diameter and 4 cm. in height and free to rotate about the shaft. The rotor is provided with 4 equally spaced bored receptacles, 3 cm. deep, to carry snugly 4 of the test vials used in the potash determination. When in operation the rotor travels within an elliptical wooden guide, the latter parallel with the plane of the flywheel and situated 11 cm. below it. The inner sweep of the elliptical guide opening measures 22 cm. short diameter and 25 cm. long diameter. It forms the upper bracing of the lower deck, the machine being supported below it by 4 corner legs. In operation the rotor turns in a direction counter to the flywheel, its orbit following the elliptical guide, contact with which causes its rotation about the shaft. The net effect is a distinct oscillatory, rocking motion impressed on the liquid contents of the test vials which simulates the desired rotary stirring action as conducted by an expert in this exacting operation. The flywheel is driven at 78 r.p.m. and the duration of rotation is fixed at one-half minute.

The Potash Rotator has been found entirely satisfactory and during the 2 years of its existence, 115 instruments have been manufactured and put in service. Following the rotation of the test solutions, the vials are transferred to the Potash Illuminator for the final reading, details of which will appear later.

The Determination of Phosphate in Soil

Following the introduction of the Potash Rotator attention was directed to improving the technic of the soil phosphate determination. The objections which still obtained, even in the modified kit procedure for the determination of soil phosphate were in some degree due to the peculiarities of the average Hawaiian soils. Organic matter, and in some cases appreciable amounts of coral brought into solution in the extraction of the soil, resulted in the development of such high discoloration or chemical distortion in the extracted soil solution that the original or modified kit method could be used with success only with soil types which did not contain objectionable constituents.

An attempt was made, therefore, to develop an entirely new rapid analytical procedure which could be employed with assurances of reliability and accuracy in all prevailing Hawaiian soil types. The final conclusion of this research gave us an entirely satisfactory and foolproof method. A detailed description of the method will be found later in this paper. The objections to the kit procedure were overcome by extracting the soil specimen with a new, weakly acidified reagent, destroying dissolved organic matter by nitric acid, converting carbonates and bicarbonates to non-interfering chlorides and redissolving the treated soil extract residue with appropriate reagents, thereafter proceeding with the analysis. The unavoidable solution of iron compounds from a few soils results in the formation of a pale yellow coloration which is carried through all the stages of the analysis. When this condition is met with, the persistent yellow tinge in the test solution blends with the blue color developed in the test and turns the normally pure blue to a shade of

green. As a compensating expedient we introduced additional series of blue-green color standards which have been found quite satisfactory in meeting the difficulty.

A Central Depot for Reagents and Apparatus

The improvements to the potash and phosphate determinations were reflected in a greatly increased expansion of plantation study of soil matters.

In December of 1934 the author recommended that all users of the rapid methods agree to employ reagents and special apparatus originating in a single source of supply. This suggestion met with plantation and Experiment Station approval. A building on the Experiment Station premises was fitted up as an R.C.M. annex. Here reagents are manufactured, aged, analyzed, tested, packaged, and stocked for distribution to all plantation laboratories of the Association. Apparatus, accessories, and special equipment used in R.C.M. work are also stocked in this building and shipped as they may be required.

The advantages of this arrangement were manifested from the very first. All members of the Association would employ reagents of uniform composition and quality. The costs of preparation were markedly reduced by handling large volumes of chemicals and supplies. Several important reagents require special care in preparation, using somewhat complicated formulae. Others remain stable only when stocked in paraffined glass containers, while still others slowly decompose after preparation and must be exchanged or renewed at 4-month intervals.

A very important factor related to the central depot arrangement for reagents embraced the matter of the checking, by Experiment Station men, of duplicate specimens of key soils which had previously been analyzed on the plantations. This checking system will be discussed later, it being mentioned here merely to illustrate the advantages accruing to the general Hawaiian R.C.M. enterprise in having a uniform and standard complement of reagents from which all workers secure their supplies.

Classes of Instruction at the Experiment Station for Plantation Analysts

As increased numbers of plantation personnel took up R.C.M. studies, a need arose for instituting courses of instruction in the work with practical laboratory analyses included as a part of the training in the technic of R.C.M. analyses. In November of 1933 the first group of 30 men attended classes held at the Experiment Station in Honolulu. The staff of the chemistry department, cooperating, Messrs. Yuen, Hamamura, Nishimura and Chu contributed their services to the attending class which was divided into small groups. In this manner it became possible to offer individual and personal instruction and assistance to each visiting student.

The centralized instruction idea met with immediate response. Classes were held thereafter at frequent intervals. They have been continued at irregular periods as the need has arisen. At present special classes are conducted for the instruction of experienced analysts in newer developments of R.C.M. and regular sessions offering general instruction are continued at occasional intervals.

Supplementing R.C.M. Studies of Soil Potash and Phosphate With New Analytical Assemblies Designed to Expand the Field of Plantation Research

Satisfactory progress having been made in correlating R.C.M. data with similar findings obtained by independent means gave impetus to the extension of R.C.M. analyses to include the determination of all of the more common nutrients not only in soils but in the cane plant, in crusher juice, in mill by-products, and in irrigation waters. Simultaneously with the development of new analytical assemblies improvements, modifications and additions were made to the older ones. For instance, in the determination of soil phosphate the procedure was adjusted and an additional complement of color standards was prepared which enabled the plantation worker to ascertain with a good degree of accuracy the amounts of available phosphate in soils in which reserves of this nutrient greatly exceeded the ranges usually found. As a matter of fact, reliable information as to the quantities of phosphate in some lands, particularly very large excesses of this nutrient, was not definitely mapped or recorded until after plantation agriculturists had completed their first surveys.

Encouraging progress in correlations suggested a resumption of crusher juice analyses for potash and phosphate. Accordingly R.C.M. assemblies were perfected which enabled an analyst to determine, with more than average laboratory accuracy, the potash and phosphate in samples of crusher juice in less than 10 minutes for both determinations. As nitrogen studies were adopted in the soil work a very satisfactory method was developed for the determination of total nitrogen in crusher juice. To augment the employment of R.C.M. data on plantations, directions for procedure and accompanying data sheets were revised to include percentage and pounds per acre-foot data for determined soil nutrients.

Plantation and Experiment Station Check on Analytical Technic

Within a period of less than two years a large majority of Hawaiian plantations had adopted R.C.M. studies, had built agricultural-chemical laboratories and had placed the routine detail of analysis in the hands of operators who had been trained at the Experiment Station in Honolulu. As a rule the responsibility of the work, the correlation of data, and field application of findings were in the hands of the plantation agriculturist.

It was apparent that the value of the study and the reliability of the findings depended entirely upon the representative value of the soils and other specimens selected for analysis and upon the accuracy, honesty, and proficiency of the operator conducting the routine analyses. The matter of correct soil sampling and adequate field survey—the vital prerequisites to any analytical study of agriculture—embraces a field of research which is receiving the attention of all those engaged in R.C.M. work. In this paper no further comment will be presented on the details of sampling research, now engaging our attention, because the purpose of the article is essentially an attempted exposition of the R.C.M. fabric. However, the reliability of the work of the plantation analyst is a matter the importance of which cannot be over emphasized. There are several factors which may contribute to faulty chemical analyses. Among these may be mentioned the condition and quality of reagents, inadequate laboratory facilities and improperly adjusted analytical aids,

such as apparatus, color standards, etc. The manner in which these hindrances to analytical proficiency have been controlled will be discussed elsewhere under "Co-operative Features of the Enterprise."

Under the above heading the attention of the reader is directed to a consideration of the important matter of the technic of the R.C.M. operator. Technic of analysis, whether it be good, indifferent or bad, is in the control only of the analyst himself. Any analyst, no matter how expert he may become, is prone to depart from conventional procedures in performing tedious routine and to inadvertently introduce personal modifications in his work. Although they may be slight, nevertheless, errors may develop which may in time become progressively serious, especially where correlations are attempted on a wide scale and interpretation of data obtained by different workers is contemplated. Other operators may become careless, listless or indifferent. Although exceedingly rare, still others may "presume," or to put it mildly, readjust or anticipate a given set of data. A great number of causes or reasons may be responsible for an analyst's tendency to anticipate data. It is equally true that anyone performing routine work, any portion of which will be subjected to analytical check by an independent operator, will give his work the very best he has in him, providing it is his desire to continue on the job.

The author, having discussed this subject with plantation managers and agriculturists and with the analysts themselves, and having received their endorsement, proposed the introduction of a cooperative checking scheme which was adopted in the early part of 1934. The plan of the checking arrangement was quite simple: the plantation analyst would set aside duplicate soil or other specimens which he had analyzed and forward them to the Experiment Station with his data; his results would then be checked with those obtained by Experiment Station analysts and the two sets of data would be remailed to the plantation with constructive comments or recommendations. The value of this feature of the R.C.M. work has justified the pains taken by the analysts to have their data independently checked. When major discrepancies are found the portions of the plantation specimens analyzed at the Experiment Station are returned to the plantation for reanalysis by him. If the second analysis on the plantation does not reveal the cause of the discrepancy then someone from the Experiment Station visits the plantation laboratory and rechecks the questioned data with the analyst in his own laboratory. The visiting Experiment Station worker remains with the plantation analyst until the causes of the discrepancy are ascertained and corrected. This checking system is now an active part of the plantation-Experiment Station cooperation, and it has proved quite beneficial to both participants.

At the present time (August 1936) we have under consideration the recommendation that beginning in 1937 all checking analyses shall be made in the plantation laboratories with the R.C.M. analyst by Experiment Station technicians. This plan, if approved, would terminate the one now in force whereby analyzed specimens are sent to the Experiment Station in Honolulu for checking analyses. It is believed that the advantages of such a modified checking system would be many. The greatest gain, we believe, which should develop from the change would be an immediate straightening out of analytical or other difficulty encountered by the plantation analyst coincident with a failure on his or our part to check his previous

work. Under the present checking system, proficient as it is, such a personal and satisfactory cooperative check is obviously out of the question.

Cooperative Features of the R.C.M. Enterprise

The inauguration of the checking system for plantation analysts led to the development of an organized cooperative plan of periodic plantation visits by an Experiment Station chemist accompanied by an R.C.M. technical expert. In the early experiences gained in visiting plantations in connection with checking the technic of the analysts, it was found that occasionally reagents had deteriorated by exposure to direct sunlight or from other causes. Perhaps color standards had faded slightly and the fact would not have been realized by the operator. Apparatus may have gotten out of adjustment and hence required realignment or other repair. On the other hand, improvements may have occurred in the plantation manner of pursuing research, for example, in placing soil-sampling stations in the field, in collecting and preparing specimens for analysis and, not uncommonly, in a marked improvement in analytical technic. Faulty reagents or apparatus, of course, required and received immediate attention at the time the plantation visit was made. But equally as important to the enterprise as a whole was the assembling and dissemination to the other plantation and Experiment Station workers of the advancements in the work which accrued as a result of research studies by distantly situated plantation men. An advantage was to be gained, we believed, if one or more persons would circulate among, and keep in closer contact with, the plantation staffs and at the same time attend to the checking system of plantation analysts and offer periodic servicing of all R.C.M. equipment, reagents, and color standards in the laboratories conducting this work. These ideas developed later into tangible form, as will be shown.

Cooperative endeavor at the Experiment Station on R.C.M. researches embraced studies under way in almost all departments. The Director encouraged this move. Chemistry, agriculture, Mitscherlich division of agriculture, pathology, genetics, and sugar technology departments participated in the work. The Island Representatives of the departments of agriculture and genetics actively participated not only in R.C.M. studies on Experiment Station projects, but extended their activities to plantation cooperation.

With the approval of the executives of the Association and the plantation managements, a visiting schedule was organized in the fall of 1934 whereby an Experiment Station chemist would call regularly at each plantation at intervals of 4 months, or more frequently. The chemist was provided with an automobile stocked with a complete complement of all R.C.M. equipment and supplies. Color standards, extra supplies of reagents, special testing apparatus, and kindred sundries were included in the car in amounts sufficient to replace any defective apparatus, reagents or other essentials which might be required by all laboratories engaged in the work. The automobile was shipped on the same steamer which carried the visting chemist on his mission.

One day or longer, usually one day, would be utilized by the visitor in discussing the work with the plantation manager or agriculturist. This feature of the visit enhanced the contacts between the plantation and the Experiment Station in addition to bringing out subjects for discussion relevant to R.C.M. and other matters

of material interest. Before the day's call was concluded the laboratory operators would be visited and their analytical technic would be checked if they so desired. Repairs or adjustments to special apparatus were made when necessary, all color standards were renewed and replacements made of any reagents which might have become contaminated, overexposed or have shown other evidences of deterioration.

As the plantation laboratories grew in numbers and as the analytical staffs were augmented to carry on additional studies using newly developed R.C.M. assemblies, it became difficult for a single visitor to accomplish all the laboratory detail in a one-day call and also engage the plantation staff in discussions on the progress of work. Then, too, details pertaining to correlations of data were demanding more and more attention as plantation surveys were completed and newer ones inaugurated. To allow the visiting chemist more time to discuss R.C.M. matters with the plantation staffs, it was arranged for a technical expert to accompany the chemist. The laboratory detail on the plantation was then taken over by the technician.

The Organization of R.C.M.

The Director of the Experiment Station, Dr. Lyon, is the administrator of the Experiment Station R.C.M. organization and of the plantation activities of the Experiment Station staff.

In January 1936, Mr. Davis, associate chemist, took over the responsibility of attention to details coincident with the operation of the R.C.M. division of the chemistry department in Honolulu. Mr. Davis, senior member of the department research staff, also devotes a portion of his activities with the author and with Messrs. Yuen, Hamamura, Nishimura, and Chu to research problems leading to improvements and modifications in existing studies and in the development of new assemblies. Francis Fong and L. Kawamura are research assistants in the laboratory studies. C. Danner and A. Postl are engaged in stockroom and in shipping duties.

The author continues in the regular duties of department head, devoting about one-half of his time to plantation visits and to research studies in the field. Assistance in plantation visits is provided by Messrs. Hamamura and Nishimura, R.C.M. technicians.

In plantation field research the author cooperates with R. J. Borden, Agriculturist at this Experiment Station and with the four Island Representatives of the Agricultural and Genetics Departments—F. C. Denison, Oahu; O. H. Lyman, Hawaii; John W. Anderson, Kauai, and D. S. Judd, Maui. Further cooperation is offered by this group to the plantation staffs in field research and in associated problems. W. L. McCleery, Sugar Technologist, J. P. Martin, Pathologist, Dr. A. J. Mangelsdorf, Geneticist and their respective staffs also contribute to the progress of R.C.M. studies.

The Chief Clerk of the Experiment Station, A. R. Grammer, and his staff, F. D. Kennedy and F. W. Littlejohn, superintend business details. Office duties in the chemistry department which are associated with the R.C.M. organization are in charge of R. Boyen.

Correlations

The value of R.C.M. data is greatly increased by correlation with other independent means of determining the status of soil fertility and by correlation with

crop response and crop requirements. At the meetings of the International Congress of Soil Science (4), held in Oxford, England, in 1935, this subject received attention of several delegates at informal gatherings. The conflict of field experiment versus chemical analysis of soil was shown to be entirely out of line by some of the members, chiefly because no conflict should exist. Either means of soil evaluation it was believed, possessed merits and advantages not found in the other. Either method of arriving at nutrient requirements for any given agricultural land might at least supplement the other, it was agreed.

In our experiences in Hawaii we have found that a site selected for a field experiment or a Mitscherlich sampling station may be totally at variance with the average fertility of what appeared to be a very uniform portion of the field.

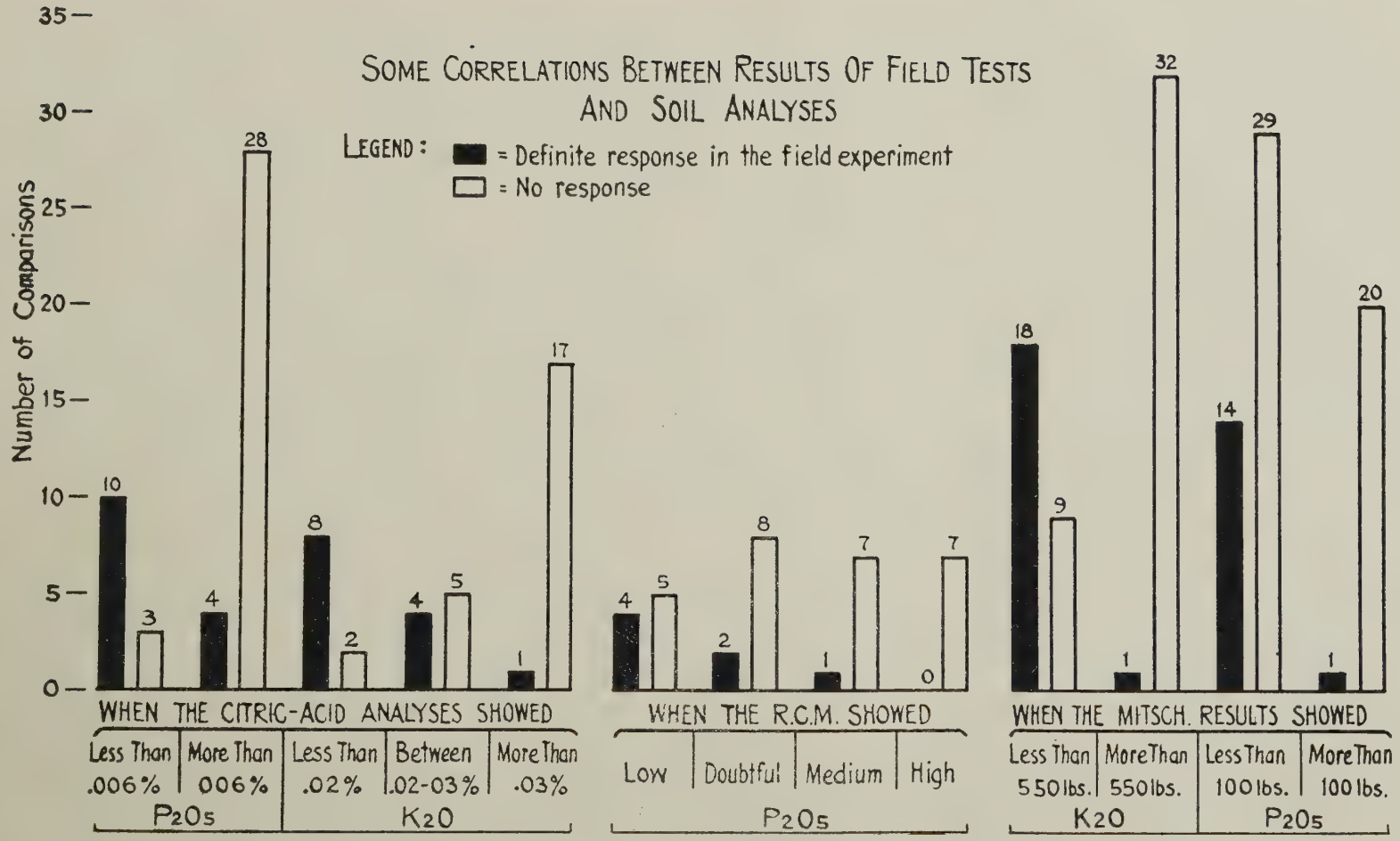
Due to the brevity of performing R.C.M. analyses and to the ease and rapidity with which a detailed survey may be accomplished in a field, an R.C.M. field study may prove a valuable preliminary step in selecting a representative location for a field experiment or Mitscherlich sampling station.

Another point in favor of correlating R.C.M. with other field studies embraces the value to accrue later to the agriculturist after such relationships have been established. A field experiment requires for completion a period equal to the age of the crop at harvest. The minimum time required for completing and reporting a Mitscherlich test is about 4 months. An R.C.M. survey may be completed in the course of a few days—certainly between harvest and the preparation of a field for the next crop (ratoon or plant). As to the wisdom of employing correlations rather than any single means of evaluating the supply of available soil nutrients in a field, we present the following paragraph by Mr. Borden:

> It is our opinion that it would be unwise to build up a plantation fertilization policy on the analytical data secured from any one single measure of soil fertility. The present extensive application of the data secured from the rapid chemical method of testing soils has been made possible only through the weight and reliance placed upon the correlating of other independent means of evaluating the available nutrient status of our soils. To dispense with methods for verifying an opinion based on results from a single type of analyses, would not be in accord with the conservative policy of the Hawaiian sugar planter.

This subject has been discussed at some length in *Handbook of Hawaiian Soils* (5). Quoting from this publication:

> Earlier studies by Stewart and McGeorge of the results of their citric acid soluble analyses of soils when compared with a response to phosphate and potash fertilizers applied in the sugar cane field, led them to suggest that (*a*) a response to applied phosphate fertilizers would not be expected if the citric soluble phosphate was above .004 per cent except on the more acid, upland soil types where the limit might be set a little higher, perhaps at .006 per cent, and (*b*) when dry land soils showed .04 per cent potash and wet land soils .05 to .06 per cent potash, little response would be obtained in the field from potash applications.
>
> A summary of data from the more recent cooperative field experiments studied at the Experiment Station, H.S.P.A., would indicate that the above phosphate figures may still hold, but it suggests a slight change in the potash limits (Fig. 12). [See illustration on page 214.]
>
> From forty-five recently harvested phosphate experiments, which are quite clearly interpretable as showing either a response or no response to phosphate applications and which are rather widely distributed throughout the Islands, we note the following correlation with citric soluble analyses of soils taken from the experimental areas:
>
> (*a*) Thirteen soils with less than .006 per cent P_2O_5, ten of which showed a response to phosphate; ten of these thirteen soils with less than .004 per cent P_2O_5, eight of which showed a definite response to phosphate.



(The above illustration is Fig. 12 from the *Handbook of Hawaiian Soils,* Association of Hawaiian Sugar Technologists, page 93, 1935.)

(*b*) Thirty-two soils with citric soluble P_2O_5 at .006 per cent or more, only four of which showed a response to phosphate applied.

From thirty-six good potash experiments from rather widely distributed cane areas, we note the following:

(*a*) Of six soils with citric soluble potash above .04 per cent, all showed no response to applications of potash fertilizer.

(*b*) Eleven soils with between .03 and .04 per cent K_2O, only one of which responded to applied potash.

(*c*) Nine soils between .02 and .03 per cent K_2O, only four of which responded to potash.

(*d*) Ten soils with less than .02 per cent K_2O, eight of which showed a definite response to potash.

The amount of data for a study of the correlation between the recently adopted rapid chemical analyses for soil nutrients, with the results secured in field experiments, is inadequate for the purpose of drawing definite conclusions at this early date. However, the data we have from thirty-four phosphate tests may be indicative of what we may expect:

(*a*) Nine soils showed *low* phosphate by the rapid chemical method (R.C.M.) and only four of these have responded to phosphate.

(*b*) Ten soils showed *doubtful* phosphate by the R.C.M. and two of these have responded to phosphate.

(*c*) Eight soils showed *medium* phosphate and one of these responded to phosphate.

(*d*) Seven soils showed *high* phosphate and none of these gave a response to applied phosphate.

Only nine potash experiments have been harvested, for which we have R.C.M. potash, and the results are not adequate for further discussion.

The correlation between the results of the Mitscherlich test with Sudan grass, and the response secured in the field experiment with sugar cane, are also indicated in Fig. 12. The data have been grouped into two divisions which indicate that either a definite response has been secured or that no response was obtained in the respective field test from which the soil was taken for the corresponding Mitscherlich analysis.

If we set our critical limit for potash at 550 pounds per acre-foot, we infer that we should get no response on soils that have more than this amount by the Mitscherlich measure, and conversely that we should get a definitely increased yield response on soils which have less than this critical limit. With this criterion, we note that our potash correlation is quite favorable, especially as regards a lack of response in the field when the Mitscherlich test has indicated more than 550 pounds of potash per acre-foot. In fact, it appears quite clear that we can reliably detect the soils that have an abundance of potash and upon which further application of potash fertilizer will hardly be justified. Our ability to find the low potash soils is not quite as good, but most of our disagreements come from the more alkaline soils and are probably due to some chemical change or pathological condition that operates differently in the Mitscherlich pots than in the field.

Setting up our critical phosphate limit at 100 pounds per acre and expecting thereby that soils with more than this amount will probably not respond to applied phosphate, and conversely that soils showing less than 100 pounds will show yield gains when phosphate applications are made, we find our data show a close agreement with the first expectation but not with the second. There are probably good reasons for this poorer correlation:

(*a*) *Pythium* root rot has been a factor in reducing yields in those Mitscherlich pots which have not received heavy applications of calcium phosphate and the low yields of these pots have undoubtedly been interpreted as due to low phosphate availability in the soil being tested.

(*b*) The indicator crop used may have been unable to extract its phosphate from a high phosphate fixing soil with sufficient speed to meet its needs during its short, rapid growth period in the pots.

(*c*) Frequently a field test may fail to show yield differences, due to plant food variations when some other limiting factor, such as drought, dominates the growing conditions in the field.

(*d*) Finally, the soil sample that was tested in the Mitscherlich pot may not have been a truly representative one. However, the encouraging feature of the phosphate correlation is the

fact that the Mitscherlich test can and does quite safely indicate where we may leave off phosphate fertilizers without reducing the cane yields.

With pineapples, Magistad had found that where there is 500 pounds per acre of replaceable potash in the soil the crop will not respond to potash fertilization. In 19 pineapple field experiments, 14 soils with more than 500 pounds of replaceable potash failed to show a response, while 5 soils that had considerably less (in fact less than 250 pounds) did respond to applications of potash fertilizer. Also, where soils have shown more than 25 parts per million (about 65 pounds per acre-foot) of readily chemical-soluble phosphorus, no response has been secured from phosphate fertilizer applied in the field.

Correlations with crops other than sugar cane and pineapples have not been definitely shown because the data are as yet inadequate.

Some correlation studies have been made between the various chemical methods for determining availability and also between chemical and biological methods. In general, the agreement has been quite good. It is doubtful, however, if the comparison of one method with another has the same value as the comparison of each method with the actual results secured in the field. Hence these methods' interrelations will not be discussed.

However, in the Director's Monthly Report for December 1936, Mr. Borden points out the relationships which appear to exist between soil pH and available nutrient supply. Included in these correlations are also R.C.M. data. Mr. Borden states:

Correlation studies from results of our 1935 soil tests at the Mitscherlich department have been made to show the relationship that apparently exists between the pH of the soil and its available supply of mineral plant nutrients. Since rapid chemical analyses were also made on these soils, our study includes both the R.C.M. and Mitscherlich measures of availability. A summary is offered as follows:

RELATIONSHIP OF SOIL pH AND AVAILABLE POTASH

No. of Tests	pH Grouping	Avg. Lbs. K_2O per acre-foot by Mitscherlich	No. of Tests	R.C.M. Class	Avg. pH
42	5.0-5.4	320	94	Low	5.5
61	5.5-5.9	554	51	Doubtful	5.9
62	6.0-6.4	640	89	Medium	6.2
60	6.5-6.9	720	25	High	6.2
19	7.0-7.4	810			

RELATIONSHIP OF SOIL pH AND AVAILABLE PHOSPHATE

No. of Tests	pH Grouping	Avg. Lbs. P_2O_5 per acre-foot by Mitscherlich	No. of Tests	R.C.M. Class	Avg. pH
42	5.0-5.4	46	36	Low	5.7
61	5.5-5.9	96	50	Doubtful	5.8
62	6.0-6.4	153	29	Medium	5.9
60	6.5-6.9	174	144	High	5.9
19	7.0-7.4	264			

The data as summarized indicate that the lower available supplies of both potash and phosphate are found associated with the more acid soil conditions.

The following article, "Soil Tests," is contributed by Mr. Borden. It is particularly appropriate to the important subject of correlations.

Soil Tests

A large share of the present activities of a plantation agriculturist is concerned with the determination of the status of available plant nutrients in the soil, so that he may recommend an optimum fertilizer practice for fields, and even for parts of fields, that are being cropped. Considerable time is being devoted to sampling and to analytical work on these samples. The rapid chemical methods for analyzing soils, crusher juices, irrigation waters and plant materials are now a part of the routine procedure concerned with a field control of fertilization at several plantations.

The question that arises quite frequently now is, "Are these rapid chemical analyses sufficient by themselves, and can we now dispense with the biological soil tests such as the Mitscherlich test and the field experiment?" May we attempt an answer to this query?

Someone has said, "A diagnosis, like a criticism, is often the opinion of one man, and no one is infallible, however great his knowledge, experience, and up-to-dateness." The analogy is applicable in two ways here: no single test of the availability of plant nutrients in the soil is infallible; and no diagnosis will be apt to be correct every time, if it has been based on data secured by a single type of investigation and measurement.

We are expected to make a thorough study and to recommend a wholly conservative fertilization policy. Like the physician who makes his diagnosis and indicates his treatment only after he has taken both your temperature and pulse beat, and looked also at your skin, eyes, mucous membrane, and secretions; and like the steel-construction engineer who not only secures chemical analysis of his alloys, but physical measurements of density and tensile strength and even X-rays of his materials, and who then applies a test load to his job before turning it over as completed, we are in a better position to support our recommendations when they have been made both on chemical analyses of soil and of plant material grown thereon, and on the more rapid biological (Mitscherlich) tests of this same soil. Especially do we consider it advisable to test all large soil groups by the Mitscherlich test, whenever we are on the point of omitting phosphate or potash fertilizer from a large area, in order that we may verify the presence of an abundant supply which has been indicated by the rapid chemical analyses. Likewise, as a further safeguard, we would urge the installation of an occasional field test on areas that appear from our rapid chemical tests to be adequately supplied with a specific mineral nutrient. This test would be a very simple one, including plots both with and without perhaps 200 pounds of the plant food in question, installed with the idea of its being repeated through successive crops so that the first indication of an approaching deficiency in the particular soil might be secured. Thus it is quite probable that both the Mitscherlich pot test, and the simple "plant-food" field experiment, will have a real value in verifying a diagnosis of adequate phosphate and potash that has been made from results of rapid chemical analyses.

The agreement between indications of the fertility status in the soil as secured from the R.C.M. (rapid chemical method) and Mitscherlich method is not always identical; neither are the indications from field experiments and R.C.M., or from field experiments and Mitscherlich tests always in agreement, although the general

correlation is high and very satisfactory. Hence there is probably a real value in having results from *both* of the measures of fertility, when any material change in fertilizer policy is being planned. Only rarely have we found no response in the field experiment when *both* the R.C.M. and Mitscherlich tests have indicated a deficiency. However, we can cite specific instances which are exceptions as follows: (1) indications of a phosphate deficiency by the Mitscherlich test, but of an ample supply by the R.C.M. test of soil from a reliable field experiment that showed no response to phosphate; (2) indications of a potash deficiency by the R.C.M. analyses but of an ample supply by the Mitscherlich test on soil from a reliable field experiment that showed no response to applied potash; and (3) no response to phosphate in a field experiment, soil from which appeared to have a very low supply by both the R.C.M. and Mitscherlich tests. It is the occurrence of this occasional exception that warrants our belief that no one of these methods of testing soils can be given up entirely. Moreover, we have not found a single instance where a definite response has occurred in a "Grade A" field experiment when the soil analyses by *both* the R.C.M. and Mitscherlich test have indicated an adequate supply of either phosphate or potash. Thus when these two particular tests will verify each other in indicating an adequate supply, we can feel pretty safe about a field fertilization policy that omits one or both of these plant foods.

The field experiment is and, for some years to come, will undoubtedly be a very definite tool to use on those soils which have been tested by R.C.M. and found to be deficient, and it is doubtful if it can be dispensed with entirely. Because of errors connected with soil sampling, variations in climatic conditions throughout the sugar cane areas, the different plant food requirements of different varieties, and the difference in composition of irrigation waters, etc., it is a most difficult undertaking to attempt a thoroughly reliable quantitative interpretation, from either the R.C.M. or the Mitscherlich results, of the most economic amount of phosphate or potash that should be applied in fertilizers to soils which apparently have an inadequate supply. Thus, having located, by use of our quicker tests (R.C.M. and Mitscherlich), large soil areas that are deficient in one or both of these mineral plant foods, it will be essential that we install a well-planned "amounts" test thereon, in order to determine the most economic amount to apply on similarly cropped areas that have given us evidence of a similar availability of these mineral soil nutrients.

Apparently therefore, neither the Mitscherlich soil test nor the field experiment will be wholly supplanted by the rapid chemical methods of soil analyses. Rather will we use all three of these valuable measures of soil fertility, correlating their results on certain key areas, and thereafter making a much wider application of the findings from the field experiment to smaller areas which are shown by the rapid chemical tests to have a similar nutrient status. Thus, plans for a differential fertilization will be on the soundest possible basis that is available at this time.

A Discussion on Methods of Chemical Analyses

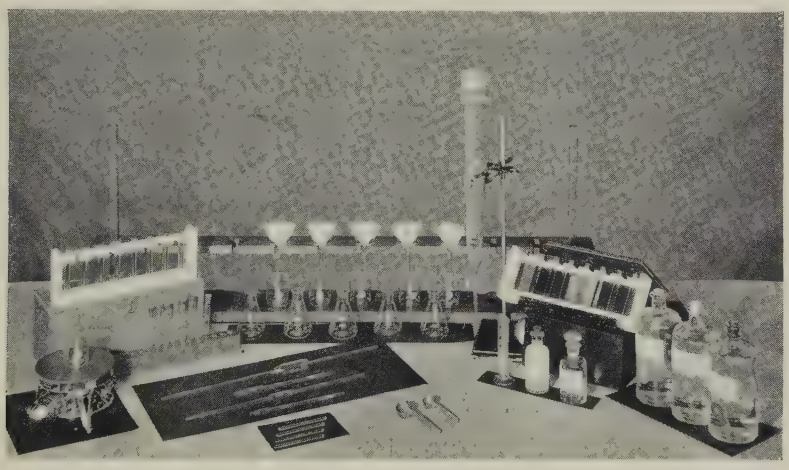
The portion of the paper immediately following is devoted to a brief exposition of the chemical methods of analysis employed in R. C. M. studies. It, in turn, will be followed with a listing of reagents by number or other designation and will include a description of the chemical composition of each reagent.

At present the full complement of R. C. M. consists of determinations of:

Available nitrogen in soil	Phosphate in boiler water
Total nitrogen in soil	Phosphate in filter cake
Total nitrogen in filter cake	Readily soluble potash in soil
Total nitrogen in mill water	Potash in crusher juice
Total nitrogen in crusher juice	Potash in molasses
Total nitrogen in plant material	Potash in irrigation water
Total nitrogen in molasses	Potash and phosphate in mill ash
Readily soluble phosphate in soil	Calcium in soil
Phosphate fixation in soil	Calcium in filter cake
Phosphate in crusher juice	Soil reaction

Available Nitrogen in Soil:

Soil-extracting mediums (acidulated salt solutions) used in the potash and phosphate determinations are not satisfactory in available nitrogen work due to the color imparted to the extracts by organic matter dissolved from many Hawaiian soils. Water alone as an extractant, without the addition of electrolyte, is also unsatisfactory



Rapid Chemical Method for the determination of available nitrogen in soils.

due to the tendency of many local soils to deflocculate when thus shaken. This makes filtration by ordinary means difficult, if not impossible. A dilute potassium sulfate solution was finally selected for extracting available nitrogen from the soil specimens.

A measured amount of soil is extracted with a definite volume of the reagent. After a maceration period of one minute, the mixture of soil and extracting solution is filtered. The filtrate is analyzed separately for ammoniacal and for nitrate nitrogen. The amount of "available" nitrogen is the sum of the two forms.

Ammoniacal Nitrogen: Five ml. of the filtrate are transferred to a 3-dram narrow shell vial for the ammoniacal nitrogen test. To this aliquot of the filtrate is added 4 drops of an alkaline tartrate solution, followed by the addition of 1 ml. of a modified Nessler reagent. The alkaline tartrate prevents precipitation in the test

EXPLANATION OF PLATE I

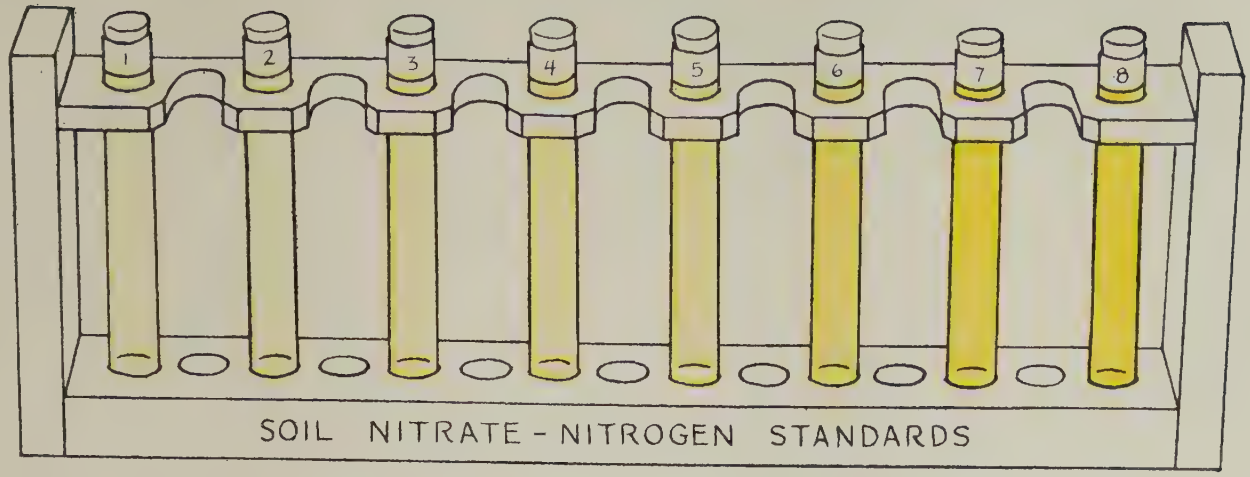
Soil Nitrate-Nitrogen Standards

The principle followed in this set of standards is similar to the others. The tubes, eight in number, increase progressively from a pale to a deep yellow. Legends appear on these tubes giving the percentage and pounds per acre-foot of nitrate nitrogen. The analyst, therefore, can jot down his findings immediately after comparing his unknown with the standard in this rack before the window of the illuminator. The numbers indicated have been placed there only for purposes of illustration and do not appear on the standards themselves.

Soil Ammonia-Nitrogen Standards

The colors increase progressively in intensity from Tube 1 to Tube 8. Percentage and pounds per acre-foot of ammonia nitrogen are indicated on each one of these standards. A more detailed discussion of all the standards will be found in the text.

PLATE I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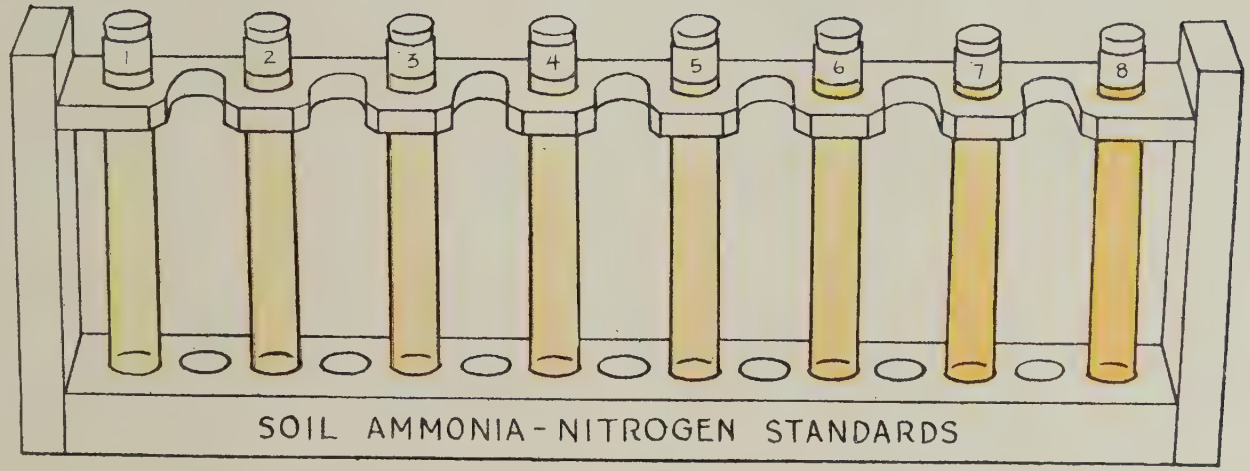
SOIL NITRATE - NITROGEN STANDARDS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SOIL AMMONIA - NITROGEN STANDARDS

vial of alkaline earths, chiefly magnesium and calcium, which may be extracted from some of our lowland soils. The Nessler reagent causes the formation of a complex compound of mercury and ammonia which is indicated by a light orange-yellow in the lower concentrations of ammoniacal nitrogen, and by darker shades for larger amounts until in the highest concentrations a brownish-orange precipitate is formed. The vial is stoppered and thoroughly shaken to disperse any precipitate that may have formed and is then matched against standards which are placed in a sliding rack mounted upon an illuminating device. The result, expressed both in per cent nitrogen (ammoniacal) and as pounds nitrogen per acre-foot, is indicated on the vials containing the color standards.

Nitrate Nitrogen: After the ammoniacal nitrogen content of the soil extract is determined, 25 ml. of the filtrate are returned to the original beaker with a pipette, after discarding the excess. This beaker containing the 25 ml. is then placed upon a hot plate and the solution evaporated nearly to dryness. Two ml. of phenoldisulphonic acid are then added to the residue and the mass disintegrated by mixing with a stirring rod. After a period of standing, to insure complete reaction of the reagent with the nitrate, the mixture is diluted with water and neutralized with concentrated aqueous ammonia. If nitrate nitrogen is present a yellow color will result, the intensity of which will vary with the nitrate content of the soil extract.

The yellow liquid is then transferred to a shell vial identical with those containing the color standards. Comparisons are then made with standards which are held in a sliding rack mounted on an illuminating device. The per cent of nitrate nitrogen and pounds nitrate nitrogen per acre-foot are expressed on the vials containing the standards.

Total Nitrogen in Cane Juice, Plant Material, Mill By-Products, and Soil:

The presence in these substances of nitrogen in organic combinations necessitates treatment more severe than that used for the determination of "available" soil nitrogen. The employment of the regular laboratory or Gunning method, without modifications, is out of the question in a scheme of rapid analysis. Costly installation of heavy apparatus makes this regular laboratory method prohibitive. It is also quite essential that a trained analyst perform the conventional laboratory determination.

Thus the problem became one of obtaining the nitrogen in the cane material in a form which could be accurately determined by R. C. M. technic with the simplest procedure and equipment. In general, when an organic substance containing nitrogen is digested with concentrated sulfuric acid at the boiling point, the carbonaceous material is destroyed and the nitrogen is converted into ammonium sulfate. After completion of this process, the solution is cooled and diluted with water. When the diluted solution is made alkaline with an excess of sodium hydroxide, ammonia is liberated. Upon heating, the ammonia is distilled into a trap containing dilute acid. Sulfate of ammonia is formed if the receiving acid medium is dilute sulfuric acid. In the usual laboratory analysis the absorption is effected in standardized acid, the excess acid remaining in the receiving flask after distillation being titrated with a standard alkali solution. In this manner the total nitrogen in the sample is determined.

The rapid method follows the principle indicated above. While in the regular laboratory procedure, cumbersome apparatus are required, the rapid method employs only equipment of the simplest design. Details have been worked out so that only a minimum of attention is required. The digesting reagent of concentrated sulfuric acid is prepared from a pure grade of material, free from nitrogen, and to it is added selenious oxide to hasten (catalyze) the decomposition of the organic sample. The usual bulky distilling and condensing apparatus gives way to an easily attached connecting bulb and tube, the latter discharging into a calibrated trap of dilute sulfuric acid cooled by a water bath. (See illustration.) Titration, too, is eliminated by simple colorimetric determination of the (ammonia) nitrogen in the dilute acid

Rapid Chemical Method for the determination of total nitrogen in cane juice, plant material, mill by-products and soil.

solution in which the distillate is collected. The entire range of the determination is adjusted so that the Nesslerization of an appropriate aliquot of the distillate gives a color falling within the limits of the series of graded ammonia color standards.

The analyst is thus relieved of the exacting detail of titration or of the cumbersome manipulation usually required in Nesslerization tests, yet without sacrifice of accuracy (as was determined in an exhaustive study). Thus is effected an appreciable saving in time and the necessity for specialized training is eliminated.

The determination of total nitrogen in soil and in dried plant material requires the use of an analytical balance where extreme accuracy is desired. With the advancement on many of the plantations to special research studies, balances have been added to the equipment. However, measured portions of soil and dried plant material, when pulverized in a mortar, may be employed in studies of total nitrogen content where comparisons are to be made of a relatively large number of samples having practically identical physical characteristics. In such cases calibrated measuring devices supplant the balance.

The determination of total nitrogen in crusher juices is made upon prepared

specimens withdrawn by standard pipettes. Fortunately the two procedures most frequently used (available soil nitrogen and nitrogen in crusher juice) require neither balance, titration nor analytical skill. Either determination may be made in one hour's time.

The procedures for the determination of total nitrogen in cane juice, plant materials, mill by-products and soils vary in details as to the handling of each individual substance (see directions). However, the principle is essentially the same in all 4 analyses. The total nitrogen determination approximates closely that obtained by the Gunning method but the procedure requires much less time and is equally as accurate.

The sample to be analyzed is digested in a 300-ml. Kjeldahl flask with a measured amount of concentrated sulfuric acid containing a mineral catalyst (selenium oxide) and a small amount of potassium sulfate. The latter is added for the purpose of raising the boiling point of the mixture. An electric hot plate of special design is used to effect the digestion. The hot acid oxidizes the carbonaceous matter contained in the sample and converts the organic nitrogen into an inorganic form. When the digestion is completed, the mass is cooled. It is then diluted with distilled water.

In the analysis of plant materials, mill by-products and soils, the solution is transferred at this point to a 250-ml. volumetric flask, cooled and made up to volume. Following a thorough mixing, a 10-ml. aliquot is removed for distillation. In the analysis of cane juice, the entire solution contained in the Kjeldahl flask after digestion and dilution is used for the subsequent distillation.

To the original Kjeldahl flask in cane juice analysis, or to a clean Kjeldahl flask containing 10 ml. of the diluted solution, is added distilled water and a measured amount of a strong solution of sodium hydroxide. The flask is then connected to a trap leading to a receiving test tube holding a very dilute acid. The test tube is immersed in a flask of cold water, which acts as a condenser. Heat is applied to the Kjeldahl flask. Ammonia, liberated by action of the alkali, is distilled over and collects in the weak acid. After distillation has been completed, the distillate is cooled and made up to a prescribed volume. An aliquot is then transferred to a 3-dram shell vial and the test completed as in the determination of available ammoniacal nitrogen in soil. The addition of Nessler reagent causes the characteristic reaction which produces the coloration in the tube. The tube is then matched with the color standards, the results being obtained by reference to a table.

Nitrogen in Irrigation and Mill Waters:

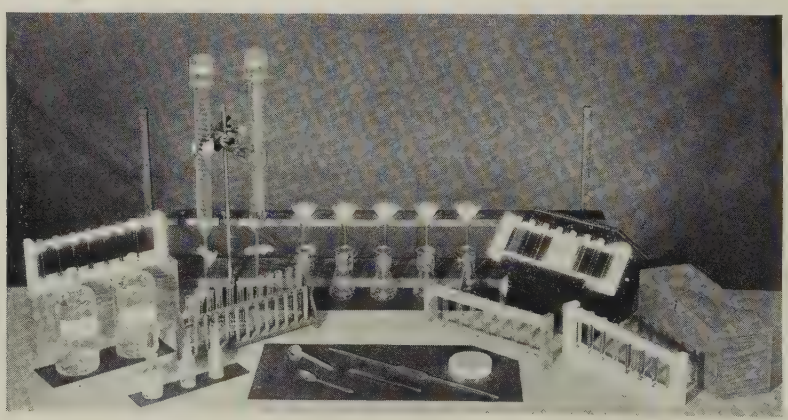
Nitrogen in irrigation water may consist of either one or more forms, ammoniacal, nitrate and/or organic. Most irrigation and drainage waters contain only ammoniacal and nitrate nitrogen. These waters may be analyzed by the procedures which follow closely the method for determining available nitrogen in soil. A sample of the water is tested directly with Nessler reagent for ammoniacal nitrogen. Another aliquot is evaporated and the phenoldisulphonic acid method is used to determine the nitrate nitrogen content.

For mill water or water to which filter cake has been added, the total-nitrogen method is employed. Concentrated sulfuric acid reagent and potassium sulfate are

added to an aliquot in a 300-ml. Kjeldahl flask. The mixture is placed on the special heater. Water is evaporated off and the organic matter is decomposed. The boiling sulfuric acid converts the organic nitrogen into the ammoniacal form. Following completion of the digestion, the residue is diluted and concentrated alkali is added. The ammonia is distilled into a dilute acid trap. The distillate is then analyzed by the regular ammoniacal nitrogen procedure. By referring to a table the p.p.m. total nitrogen and pounds total nitrogen per million gallons of water are obtained.

The Determination of Readily Soluble Phosphate in Soil:

A 10-gram portion of soil specimen from a calibrated metal cup is placed in a 125-ml. Erlenmeyer flask. To the soil in the flask is added a measured amount of a reagent which consists of dilute hydrochloric acid solution. The soil and reagent are then shaken for one-half minute and immediately filtered into a 50-ml. beaker. Ten ml. of the filtrate (soil extract) are pipetted from the beaker into another 50-ml. beaker and treated with several drops of concentrated nitric acid delivered from a dropping bottle provided for this purpose. The beaker with its contents of soil extract and nitric acid is transferred to a low-heat electric hot plate and evaporated to dryness, whereupon several drops each of concentrated hydrochloric and nitric



Rapid Chemical Method for the determination of readily soluble phosphate in soil.

acids are added and the extract again evaporated to dryness. As a result of the hydrochloric-nitric acid treatment organic substances which might otherwise interfere with the subsequent color development are destroyed and a light colored residue is obtained. The beaker containing the residue is cooled and to it are added 10 ml. of a reagent consisting of a carefully prepared dilute hydrochloric acid solution of ammonium molybdate. When the residue is dissolved by the reagent, the solution is transferred to a tall, narrow, phosphate comparison tube. Two drops or more of a reagent consisting of stannous chloride dissolved in dilute hydrochloric acid are added to the tube containing the test solution, whereupon it is well mixed by closing the tube with the thumb and inverting the tube several times. Immediately following the full development of the blue color resulting from the reaction between the

stannous chloride solution and the phosphate molybdate compound, the tube is transferred to an open groove in a sliding rack which holds a series of color standard tubes. This rack is mounted on an illuminating box devised for this type of analysis. The intensity of the color developed is approximately proportional to the amount of phosphate extracted from the soil. The color tube is compared with standards which are marked "High," "Medium," "Doubtful," and "Low," and the results are recorded accordingly. By referring to a data sheet which accompanies the assembly, one may obtain the percentage of P_2O_5 in the soil specimen or the concentration in terms of pounds of P_2O_5 per acre-foot of soil. In addition to the above-mentioned set of standards, another series is provided for the estimation of amounts of P_2O_5 greater than that indicated by the "High" standard represented in the regular color standard set.

The Estimation of Soil Phosphate Fixation:

This procedure is used in order to determine the relative degree to which soils are capable of absorbing applied soluble phosphates. A phosphate solution of def-

Rapid Chemical Method for the estimation of phosphate fixation in soil.

inite concentration is shaken with a prescribed amount of soil and filtered after a specified period of contact. The amount of phosphate remaining in solution is indicated by the intensity of the blue color developed by the reaction of phosphate with the ammonium molybdate and the further reaction with stannous chloride. The resulting blue color is matched against standards contained in sealed vials and the measured degree of fixation is expressed in arbitrary numerical indices from 0 to 90 to which are prefixed the serial numbers of the phosphate fixation solution used in the tests.

The fixing solutions, adjusted to neutral reaction, contain 100, 500 and 1000 parts per million phosphate and are designated respectively as solutions Series 100,

EXPLANATION OF PLATE II

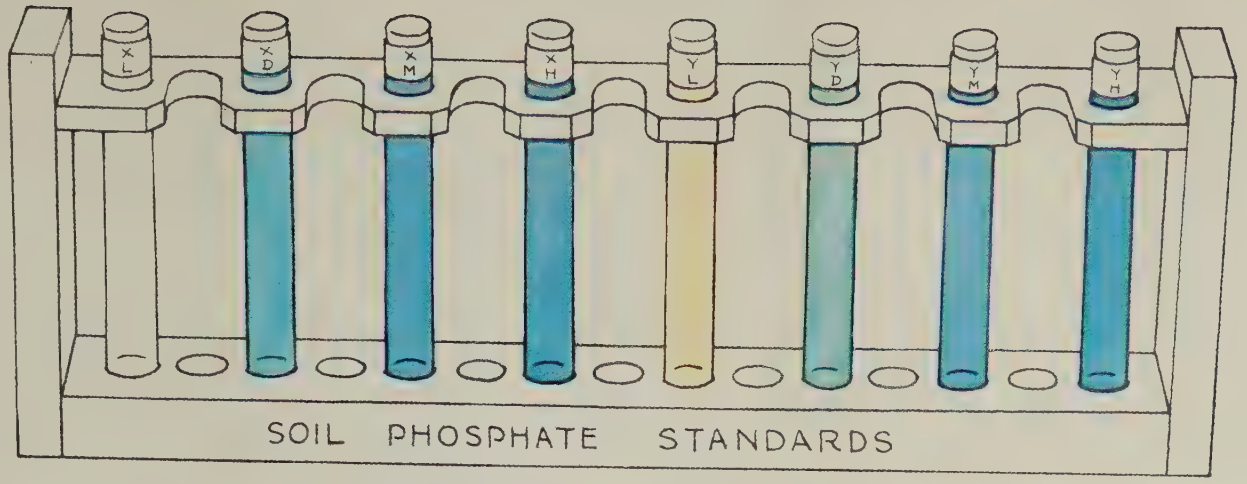
Soil Phosphate Standards

The tubes shown are placed in the stationary positions in the rack, the unknown samples being compared in the notched intervening spaces. The legend "X" indicates a type of blue coloration which may be developed in normal or water-white soil extracts. The legend "Y" indicates a green-like blue coloration which is produced in the analysis when the soil extract is somewhat yellow because of ferric iron which it may contain. The letters "L," "D," "M" and "H" represent, respectively, low, doubtful, medium and high concentrations of the nutrient.

Cane Juice Phosphate Standards

The numbers refer to a progressively increasing depth of color produced in the test, the matching of the unknown being made by comparison in the notched spaces provided in the rack before the light of the phosphate illuminator.

PLATE II


XL
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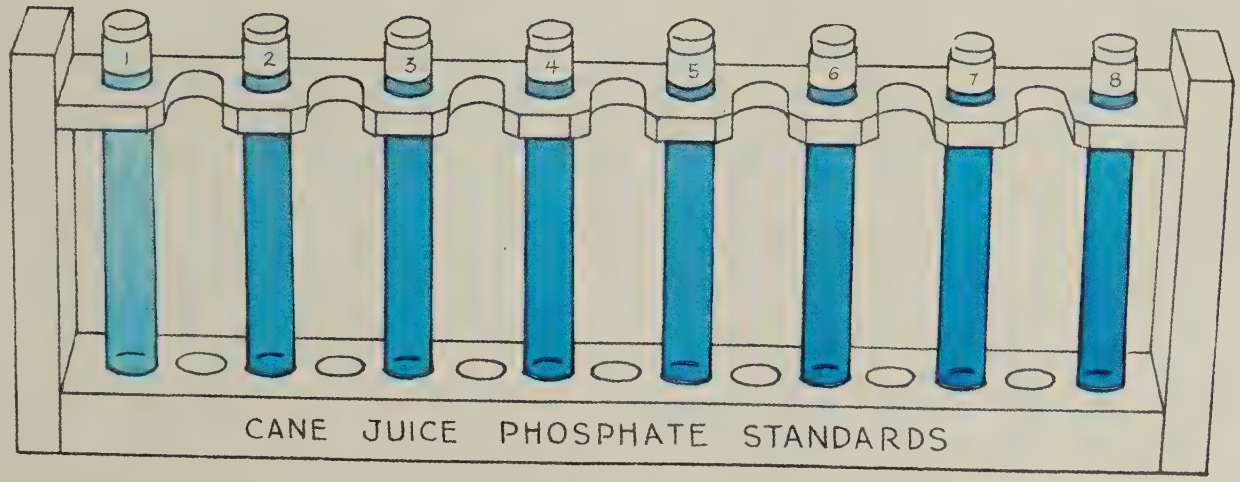
SOIL PHOSPHATE STANDARDS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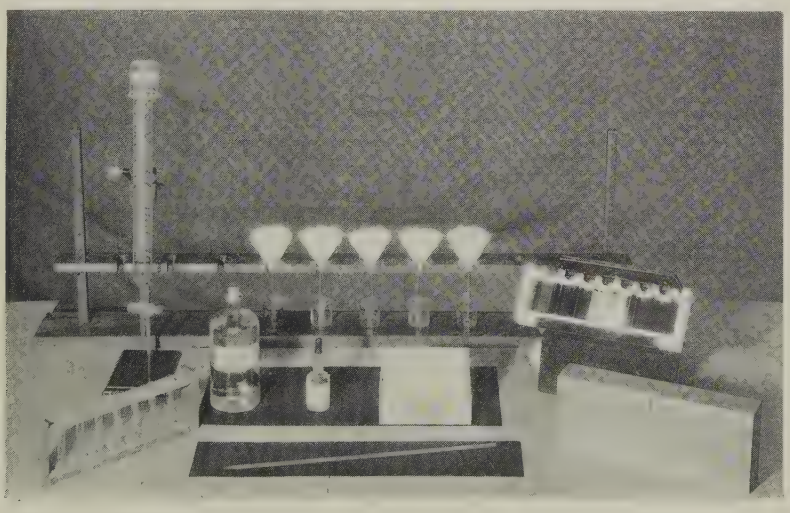
CANE JUICE PHOSPHATE STANDARDS

500 and 1000. Using these solutions at the specified ratio of 30-ml. solution to 5 grams of soil, tests may be performed equivalent to treatments with 1500, 7500 and 15,000 pounds P_2O_5 per acre-foot of 2.5 million pounds of air-dried soil. These solutions are buffered to minimize the tendency of many of our Hawaiian soils to deflocculate when in aqueous suspension.

In making the tests, 5 grams of air-dry soil are measured in a calibrated metal cup and transferred to a 2-ounce graduated bottle. Then 30 ml. of Series 100 fixation solution are added. The mixture is shaken for 1 minute and then allowed to stand for 24 hours. At the end of this period it is reshaken for another minute, then the liquid and soil are separated by filtration. The filtrate is collected in a 3-dram, tall vial until it has been filled to about two-thirds of its capacity. One ml. of ammonium molybdate is added and the solution is thoroughly mixed. Then 1 drop of stannous chloride reagent is introduced and the solution is again mixed. A blue coloration will be produced in the test solution if phosphate remains in the filtrate. This blue color is matched against the standards. If the index obtained by this test is "60" or above, fresh portions of the soil may be retested, using the more concentrated solutions.

Two 5-gram portions of soil are transferred separately to bottles containing 30 ml. each of the solutions Series 500 in one case and Series 1000 in the other. Soil and solutions are shaken for 3 minutes, allowed to stand 24 hours, reshaken for 3 minutes, and finally allowed to stand for another 24 hours. At the end of this 48-hour period the mixture is shaken for another 3 minutes and filtered. The color test for these filtrates is performed in the same manner as for the Series 100 solution. The numerical indices are determined and tabulated with the prefixes of the series numbers of the fixation solutions.



Rapid Chemical Method for the determination of phosphate in cane juice.

The Determination of Phosphate in Cane Juice:

The sample of cane juice for analysis is filtered into a clean beaker. By using a special 0.5-ml. pipette graduated in 0.1 ml., a 0.3-ml. portion of the filtered cane

EXPLANATION OF PLATE III

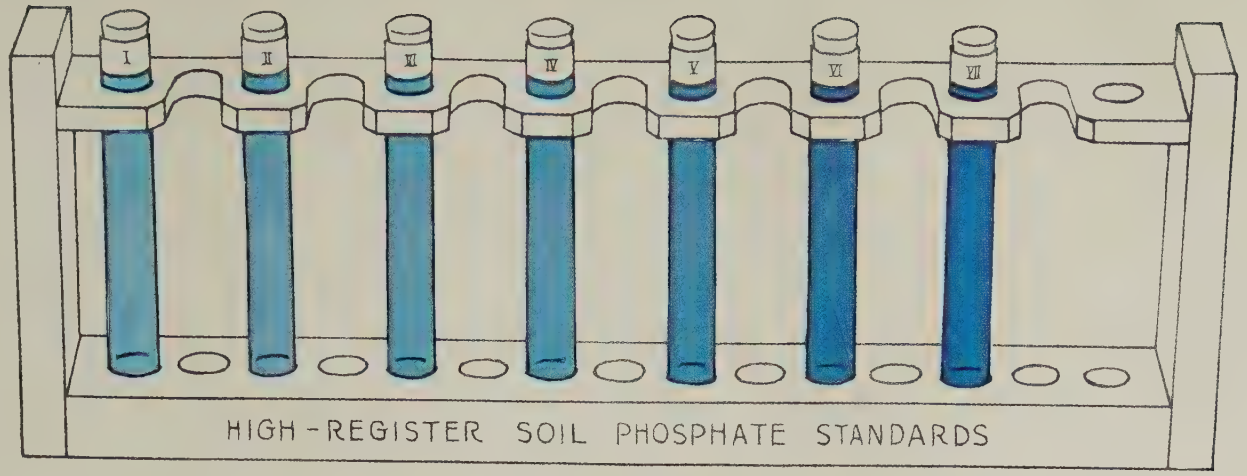
The High-Register Soil Phosphate Standards

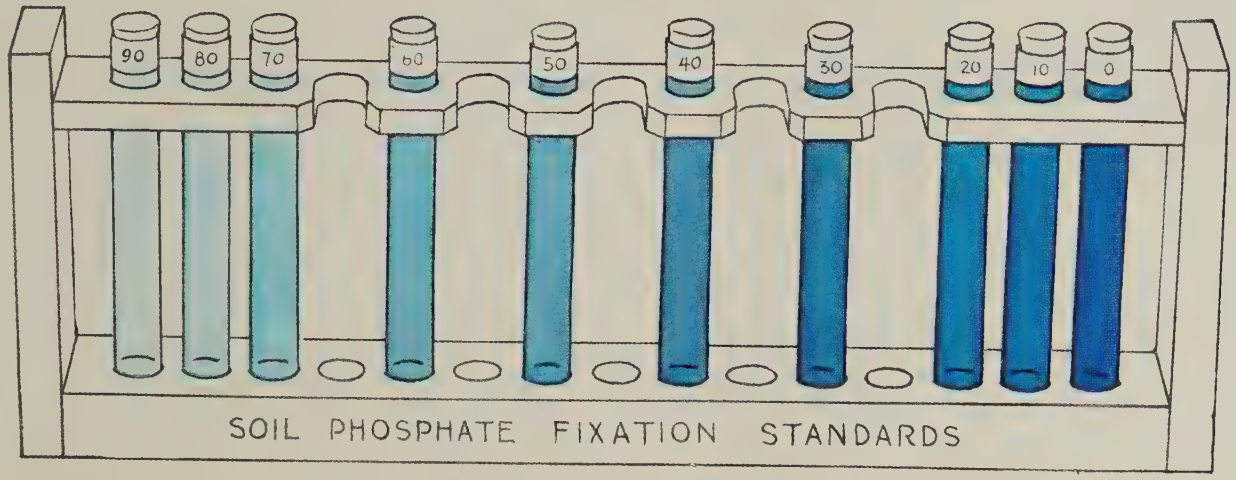
These standards were developed to enable the analyst to obtain more specific results where the concentrations of soil phosphate went beyond the limit of the "High" value in the regular standards. Here the more intense shades of blue are separated in gradual stages in seven steps and in this case the standards each carry a Roman numeral, increasing progressively with the depth of color in addition to a percentage and pounds per acre-foot figure of the corresponding concentration of phosphate as it exists in the field from which the soil was taken.

Soil Phosphate Fixation Standards

This series of color standards is divided in depth of hue of coloration, the very palest discernible blue having been given an index number of 90, the depth of blue color becoming progressively deeper until a zero is reached, the latter being the deepest color tube in the set. The reason for using 90 to indicate the faintest blue color was originally intended as an indication of fixation of applied phosphates at 90 "per cent" or higher, and a test giving a color comparable to the zero tube, or full blue, would have passed through the analysis without having any phosphate fixed. Hence, roughly, phosphate fixation would have been negligible.

PLATE III


I
II
III
IV
V
VI
VII
HIGH-REGISTER SOIL PHOSPHATE STANDARDS


90
80
70
60
50
40
30
20
10
0
SOIL PHOSPHATE FIXATION STANDARDS

juice is transferred to a phosphate comparison tube, whereupon a solution consisting of dilute hydrochloric acid and ammonium molybdate is added until a definite volume is obtained. The color is developed with 1 or 2 drops of a stannous chloride solution and compared with a set of color standards prepared for cane juice analysis. When the color developed is darker than the darkest tube, smaller aliquots of either 0.2 ml. or 0.1 ml. are taken for analysis and conversely, when the color is lighter than the lightest tube, larger aliquots of either 0.4 ml. or 0.5 ml. are treated and compared. The aliquot of juice taken and the number on the color standard with which the unknown was matched, when referred to a data sheet, will indicate the percentage of P_2O_5 by volume in the juice sample.

The Determination of Free or Soluble Phosphates in Boiler Water:

The collected sample is passed twice through filter paper to separate all precipitated phosphates from free phosphates. Two ml. of filtrate are transferred to a 3-dram, tall vial to which is added a solution consisting of dilute hydrochloric acid and ammonium molybdate until a definite volume is obtained. By the addition of 1 or 2 drops of stannous chloride solution a blue coloration is produced which is compared with the juice phosphate color standards on an illuminator. The concentration of free phosphates in parts per million is obtained by reference to a data sheet provided for this purpose.

The Determination of Phosphate in Filter Cake:

A weighed amount of filter cake is extracted with a measured quantity of a one-half normal solution of hydrochloric acid. To an appropriate aliquot of the extract, contained in a shell vial, is added a dilute acid solution of ammonium molybdate. The solution is then mixed and stannous chloride reagent, which develops a blue coloration, is added. The unknown is then matched against the color standard tubes used for the phosphate in cane juice determinations. The tube matched is referred to a table from which the results obtained are expressed as per cent P_2O_5 and as pounds P_2O_5 per ton of filter cake.

Determination of Phosphate in Mill Ash:

A 1-gram sample of mill ash is weighed on an analytical balance and placed in a 125-ml. Erlenmeyer flask. To the flask is added a measured amount of dilute hydrochloric acid. The flask is then shaken for 3 minutes and the solution immediately filtered into a 100-ml. beaker. Ten ml. of the filtrate (or extracted solution) are pipetted into a 50-ml. volumetric flask which is then filled up to its 50-ml. mark with distilled water, stoppered and thoroughly mixed. By means of a special 0.5-ml. pipette, graduated in 0.1 ml., a 0.3-ml. portion of the well-mixed solution in the flask is transferred to a phosphate comparison tube. The solution in the tube is then made up to a definite volume with a reagent consisting of a dilute hydrochloric acid solution of ammonium molybdate. One or 2 drops of stannous chloride solu-

tion are then added and the color thus developed is compared with the set of color standards used in the R.C.M. phosphate procedure for cane juice. When the color developed is without the range of the standards, a suitable aliquot of the diluted solution is taken and the comparison again made. This process is repeated, if necessary, until a good match is obtained. By referring the amount of solution taken and the number on the matching color standard to a printed data sheet, the percentage of P_2O_5 in the mill ash is obtained.

The Determination of Readily Soluble Potash in Soil:

The soil specimen to be analyzed is transferred from a calibrated metal cup, holding 2.5 grams, to an Erlenmeyer flask of 125-ml. capacity. To the flask is added a measured amount of a reagent acidulated with nitric acid. The soil and reagent

Rapid Chemical Method for the determination of readily soluble potash in soil.

are agitated for one-half minute whereupon the mixture is poured into a previously prepared filter, the filtrate (soil extract) being collected in a 50-ml. beaker. One ml. of the soil extract is pipetted from the beaker and transferred to a flat-bottom comparison tube. Four drops of a reagent, consisting of a carefully standardized solution of cobaltous nitrate and sodium nitrite in acidulated distilled water, are now added. The comparison tube with its contents of soil extract and reagent is gently agitated for a brief interval and then placed in the recess of an inclined support. Immediately thereafter 1 ml. of an alcoholic reagent is carefully introduced along the inclined inner surface of the tube, forming a clear supernatent layer above the mixture of soil extract and reagent. The comparison tube, with its contents, is then carefully transferred to the rotor of the Experiment Station electric (or spring-driven) potash rotator. As a result of the combined oscillating and rotating action of the rotor in its orbital swing, the contents of the comparison tube are subjected to a standardized and peculiarly characteristic motion which results in the develop-

ment of a turbidity, the extent of which is directly proportional to the amount of potash extracted from the soil specimen. The rotating device is operated for exactly one-half minute. The comparison tube is then immediately transferred to the slot of the illuminating instrument prepared for this type of analysis. The degree of turbidity is determined by sighting vertically down through the column of liquid in the comparison tube, gauging the turbidity by the appearance or non-appearance of a number of parallel ruled lines graded in intensity from very faint to very heavy in a luminous field over a ground glass background. The various series of parallel lines are numbered 1, 2, 3 and 4. The operator refers the number of the group of lines which he can just distinguish to a data sheet which accompanies the assembly and from which he may read off directly the percentage of potash in the soil specimen, or its concentration in the field in pounds per acre-foot.

Rapid Chemical Method for the determination of potash in cane juice.

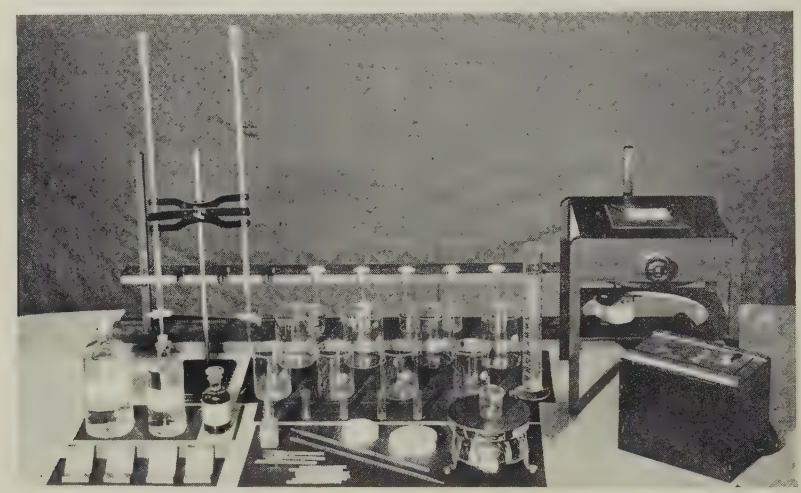
Determination of Potash in Cane Juice:

The juice collected for analysis is passed through a coarse filter paper. A preliminary test to determine the dilution necessary is conducted by adding, in a flat-bottom comparison tube, 1 drop of the juice to 1 ml. of a buffered reagent which consists of a solution of sodium acetate in dilute nitric acid. The procedure is repeated for a second vial, using 2 drops of juice. To each tube 4 drops of a standardized reagent consisting of a solution of sodium nitrite and cobaltous nitrate are added. The contents of the tubes are mixed slightly, and 1 ml. of the alcoholic reagent added along the inner wall of the inclined vial. The vials are then placed in the potash rotator and shaken for exactly a half minute. Readings are made on the standard potash illuminator. The technic following the dilution of the sample is carried out in exactly the same manner as that used in the soil potash assembly. Readings of the ruled potash chart are similarly made. For all the possible readings a table is supplied as a part of the procedure, giving the recommended dilution to be made for the regular analysis of the sample. These final dilutions are made by

pipetting out 1 ml. of the juice and adding the volume of the dilutent determined by the preliminary test. One ml. of the diluted juice is transferred to a comparison vial, the necessary reagents added, the sample rotated and the readings taken as before. The percentage of potash in the juice is read off directly from a table which accompanies the assembly.

The Determination of Potash in Irrigation Water:

A measured volume of water is evaporated to dryness in a 400-ml. beaker. The potash in the residue is taken up in 10 ml. of a solvent consisting of sodium acetate and dilute nitric acid. After thoroughly policing the beaker, the solution is filtered into a 50-ml. beaker. Fractional parts of a ml. of this filtrate are placed in potash vials and made up to a full ml. with the potash solvent. The regular potash reagents



Rapid Chemical Method for the determination of potash in irrigation water.

are then added. This is followed by shaking in the potash rotator. Readings are made on the potash illuminator in the usual manner. By referring these readings to a table accompanying the assembly, the concentration of potash is indicated directly in terms of parts per million, or pounds K_2O per million gallons. These figures are given under the proper column of potash solvent used in taking up the residue after evaporation of the water sample and the fractional part of a ml. of the extracted filtrate employed in the analysis.

The Determination of Potash in Mill Ash:

A weighed portion of a representative and pulverized sample of mill ash is treated for 1 minute with 50 ml. of a reagent consisting of sodium acetate and dilute nitric acid, and the mixture is then filtered. Fractional parts of a ml. of the filtrate are transferred to the regular potash comparison tubes and the volume made up to a full ml. with the potash solvent. Four drops of a reagent consisting of sodium

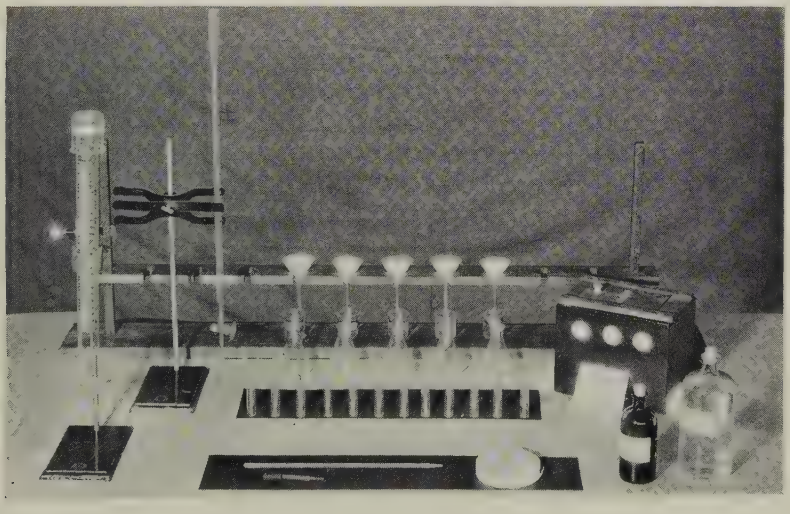
nitrite and cobaltous nitrate are then added and quickly incorporated. One ml. of an alcoholic reagent is allowed to flow into the tube with a minimum amount of mixing. The vial is then put into the potash rotator, shaken for half a minute, and the readings taken on the potash illuminator in the same manner as for soil potash. When the correct aliquot has been used the reading falls within the limits of the ruled chart. By referring to a special table, the potash content of the ash is read directly in terms of per cent K_2O.

Determination of Potash in Molasses:

A weighed sample of molasses is diluted to the point where its potash content approximates that of a representative specimen of cane juice. The solution is then filtered. It is possible to proceed from this point with an aliquot of the filtrate precisely as if it were filtered cane juice. Accordingly, the analyst is referred to the directions for the rapid estimation of potash in cane juice. A table applicable to potash in molasses has been prepared, taking into account the weight of the sample and the dilution. The results are expressed in terms of percentage and pounds of K_2O per ton of molasses.

The Determination of Readily Soluble Calcium in Soil:

Air-dried soil is transferred from a metal cup holding 2.5 grams to an Erlenmeyer flask of 125-ml. capacity. Thirty ml. of a neutral solution of ammonium acetate are added to the soil from a dispensing burette. The soil and reagent are given approximately 100 swirls in about one-half minute, and immediately poured into a previously prepared dry filter and collected in a 50-ml. beaker. One ml. of



Rapid Chemical Method for the determination of calcium in soil.

the filtrate is transferred to a short, flat-bottom comparison vial and is made up to 2 ml. with the calcium solvent. One ml. of calcium precipitant consisting of oxalic acid dissolved in dilute acetic acid is added from a calibrated dropper. The

tube containing the mixture is closed with the thumb and given 30 rapid shakes. The solution is allowed to stand for one-half minute and is then placed on the calcium illuminator. This instrument is similar to the potash illuminator in design, but contains its own standardized card of 3 sets of lines. The calcium content varies directly as the turbidity of the solution and this is measured in terms of the visibility or non-visibility of the lines on the calcium card. The readings are referred to a table accompanying the assembly which gives the calcium content in terms of per cent CaO and pounds CaO per acre-foot.

For each soil sample determinations are made with different aliquots of the filtrate until consecutive readings of both 2 and 3 are obtained. By referring to the table the lower figure represented by either one of these readings is recorded as the final result. The consecutive readings of 2 and 3 are required to select an accurate range of the calcium concentration, for unlike the rapid method for potash, usually 2 or more readings of 2 can be obtained. The lower figure indicated by the consecutive readings in the table is reported because the chart (special for calcium) has been calibrated to give readings of 2 and 3 at two definite minimum concentrations of calcium.

The Determination of Total Calcium in Filter Cake:

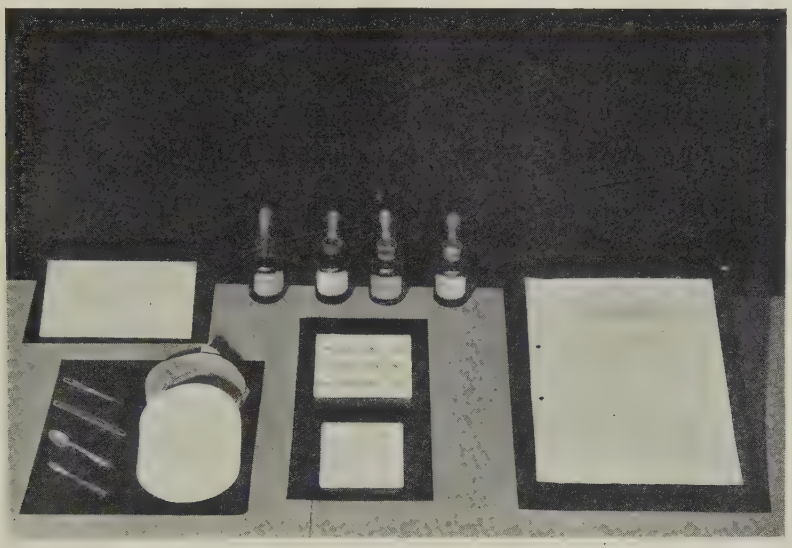
A weighed amount of filter cake is extracted with a measured volume of a one-half normal solution of hydrochloric acid. To 5 ml. of the extract is added an equal volume of 0.48 normal solution of sodium hydroxide. Successively increasing aliquots of 0.1 ml. to 1.5 ml. of the mixed solution are transferred to short shell vials. Four such aliquots are taken for each reading. The contents of the vials are made to 2 ml. with a solution of neutral normal ammonium acetate. A precipitate of calcium oxalate is formed by the addition of 1 ml. of a reagent consisting of oxalic acid in dilute acetic acid solution. The vial is closed with the thumb. It is then given 30 vigorous shakes in 7 seconds. The turbidity produced as a result of this treatment is viewed against a chart of ruled, parallel green lines in groups of increasing intensity before a background of diffused light in a specially prepared illuminating device. The illuminator slide is placed so that the slots open on the No. 3 lines of this chart. The extent of turbidity produced in this analysis is measured by the masking effect of the oxalate suspension upon the chart grouping of the darkest green lines. Three tubes are placed in the slide each time. The tube through which the lines are clearly visible is removed and the fourth tube is placed on the slide. Notation is made of that aliquot which produces a turbidity such that upon sighting through the column of precipitate the heavy ruled or No. 3 lines are just barely visible. The reading for this aliquot is "3." Then the next larger aliquot in which the lines are not visible is noted, a "4" reading. The readings are referred to a table and the results for the 2 aliquots are obtained. The final value representing the analysis of the sample is the average of the values for the 2 selected readings. The result is expressed as per cent CaO and pounds CaO per ton of filter cake.

The Colorimetric Determination of Soil Reaction (pH):

The procedure for the rapid colorimetric determination of soil reaction is sim-

ple in principle. It consists essentially in passing a suitable organic pH indicator through a small sample of soil and noting the color change of the indicator brought about by the free hydrogen or hydroxyl ions present in the soil. The final color thus obtained is compared with a color chart of that particular indicator and when a match is obtained, the pH reading is taken directly from the chart.

Preliminary tests run with an artists' porcelain spot plate serve to determine the proper indicator to be used for any particular soil and at the same time give the approximate pH readings.



Rapid Chemical Method for the colorimetric determination of soil reaction (pH).

Having determined the correct indicator for the soil under analysis, the test is repeated, using the LaMotte-Morgan pH block with which the final and more accurate result is obtained. A small sample of soil is placed in the compartment above the perforated partition and the indicator added drop by drop until the soil is saturated. Two to 3 drops more are added, and by a very light application of the sharp end of a glass rod against the perforations of the partition on the side opposite the soil, the solution is drawn out by capillary action. This solution is then drawn down the channel into the lower depression by using the large end of the rod. The color of the liquid thus obtained is immediately matched with the color chart of the particular indicator used and the pH reading taken directly from the chart.

The Reagents Used in R.C.M. Work

The numbering of reagents in the analytical directions which follow may appear to be erratic. The reasons for this are that some of the older reagents have been discontinued while others have been given numbers by adding a cipher (0) or a ten (10) to an older reagent designation in order to associate a newer modification with a much used and earlier standard solution.

A Word Regarding Reagents:

We have found by experience that the only possible manner of insuring accurate and reliable R.C.M. analyses is by centralizing the preparation of the various reagents for the whole Hawaiian enterprise in the hands of trained chemists. Sporadic and perfectly good-intentioned excursions into this field by a few untrained workers have resulted in the loss of otherwise good laboratory effort in worthless analyses. Losses of money and time and delays to execution of work have also resulted from the employment of unfit or faulty reagents. The Experiment Station assumes the responsibility of maintaining the highest possible quality in the reagents supplied from the Honolulu laboratories. The inspection service by the Experiment Station is designed, among other objectives, to maintain the high quality of reagents under laboratory usage by the plantation staffs.

Reagent No. 1, K_2O:

An aqueous solution of sodium acetate, C. P., 500 grams in 8000 ml. of distilled water. To 10,500 ml. of this solution are added 4500 ml. of C. P. nitric acid in 1-1 solution.

Reagent No. 2, K_2O:

A solution of C. P. cobaltous nitrate, 318 grams and C. P. sodium nitrite, 1200 grams in distilled water, 4000 ml. containing 100 ml. of C. P. acetic acid. This reagent must remain for a time in storage in the dark, later is filtered, tested and shipped in small, amber glass-stoppered containers. It decomposes rather readily in the tropics and requires occasional inspection.

Reagent No. 3, K_2O:

Redistilled ethyl alcohol, exactly 95 per cent, containing the denaturents pyridine and xylol.

Reagent No. 4, P_2O_5:

A solution of 100 grams C. P. ammonium molybdate and 850 ml. of distilled water—stored, aged and filtered. Seventeen hundred ml. C. P. concentrated hydrochloric acid (free from arsenic and phosphates) is added to 700 ml. distilled water. The above solutions are combined and then stored in large "non-sol" glass containers. This is the concentrated stock solution. To prepare the reagent, 120 parts of stock solution are diluted to 1000 parts with distilled water. The finished reagent is stored and shipped in parafined glass containers.

Reagent No. 5, N:

This is an approximately 0.3 normal solution of C. P. potassium sulfate.

Reagent No. 6, N:

A solution of mercuric chloride is saturated by boiling with an excess of the salt. A solution of potassium iodide is prepared by dissolving 61.75 grams of the C. P. chemical in 250 ml. of distilled water.

To the potassium iodide solution an amount of the cold mercury solution is added, just sufficient to make the color a permanent bright red. The red precipitate is dissolved by adding exactly 0.75 gram of potassium iodide. Next add a solution of 150 grams C. P. potassium hydrate dissolved in 250 ml. of distilled water. Add distilled water to 1000 ml. Allow to settle clear, age for a time, test and bottle.

It is best to make up a large amount of Nessler solution. If, by its use, the ammonia standards do not match the artificial ones prepared, a little more mercuric chloride to increase sensitiveness, or potassium iodide to decrease it, will bring the Nessler's solution to the point where, if just 1 ml. is used, the regular ammonia standards will exactly match the artificial ones. The artificial standards may then be employed for the ammonia readings until the Nessler solution tested is entirely used. Of course, each new lot of Nessler solution should be compared to see that it has the proper degree of sensitiveness to match the standards.

Reagent No. 6A, N:

Dissolve 500 grams sodium-potassium tartrate, C. P. and free from nitrogen in 1000 ml. of ammonia-free distilled water. Add 5 ml. Reagent 6, N, for each 50 grams of salt dissolved. Stir and let stand overnight. Add an additional few drops of Reagent 6, N, to detect any unprecipitated ammonium salt. If found, repeat addition of Reagent 6, N, let stand as before, and continue in this manner until no further precipitate is formed. Filter and age the solution by storing for a while. Dispense in rubber-stoppered bottles.

Reagent No. 7, N:

Dissolve 200 grams pure white phenol in 1200 ml. of N-free sulfuric acid. Add 600 ml. fuming sulfuric acid (15 per cent SO_3) and stir well. Heat for 2 hours at 100° C. Store in clean glass containers. Dispense in glass-stoppered bottles.

Reagent No. 8, N:

Distilled water free of ammonia.

Reagent No. 9, N:

C. P. concentrated ammonium hydroxide.

Reagent No. 10, K_2O:

A 10 per cent solution in distilled water of Reagent No. 1, K_2O.

Reagent No. 13, CaO:

Dissolve 1232.96 grams C. P. ammonium acetate in 16,000 ml. distilled water. Age for a few months before adjusting pH to neutrality.

Reagent No. 14, CaO:

Dissolve 32 grams C. P. oxalic acid in 1000 ml. of a 36 per cent acetic acid solution.

Reagent No. 15, Total N:

Concentrated C. P. sulfuric acid free from nitrogen, containing 0.25 gram selenious oxide per 100 ml.

Reagent No. 16, Total N:

A solution consisting of 40 ml. normal sulfuric acid diluted to 1500 ml. with distilled water.

Reagent No. 17, Total N:

A solution of sodium hydroxide, 275 grams dissolved in 750 ml. of water, boiled for 5 minutes. Cool and store.

Reagent No. 18, Total N:

Redistilled water by special treatment to render it free from ammonia and other impurities.

Reagent No. 19, CaO:

A 0.48-N solution of C. P. sodium hydroxide.

Reagent No. 40, P_2O_5:

A solution of 133 grams C. P. ammonium molybdate and 850 ml. distilled water is aged and filtered. A mixture of 2267 ml. C. P. concentrated hydrochloric acid (arsenic and phosphate free) and 133 ml. of distilled water is prepared. These solutions are combined and stored in large "non-sol" glass containers. This is the stock solution. To prepare the reagent, 120 parts of the stock solution are diluted to 1000 parts with distilled water. The finished reagent is stored and shipped in paraffine-lined glass containers.

Series 100 Phosphate Fixation Solution:

One hundred parts per million P_2O_5 from diammonium phosphate, plus 160 grams potassium sulfate per 16,000 ml. of reagent.

Series 500 Phosphate Fixation Solution:

Five hundred parts per million P_2O_5 from diammonium phosphate, plus 240 grams potassium sulfate per 16,000 ml. of reagent.

Series 1000 Phosphate Fixation Solution:

One thousand parts per million P_2O_5 from diammonium phosphate, plus 480 grams potassium sulfate per 16,000 ml. of reagent.

Stannous Chloride Solution:

A 2½ per cent solution of C. P. stannous chloride dissolved in 1:10 hydrochloric acid. Observe usual precautions of maintaining tin in stannous form.

Ammonium Molybdate Solution:

Dissolve 25 grams of ammonium molybdate in 200 ml. distilled water heated to 60° C. Filter. Dilute 280 ml. of arsenic and phosphate-free, concentrated sulfuric acid (36 N) to 800 ml. with distilled water. When cool, combine the 2 solutions with care. When again cool, dilute to 1000 ml. This is a stock solution. It should be aged in the dark. The reagent is prepared by diluting the stock solution with equal parts of distilled water.

Bromthymol Blue pH Indicator:

Pipette 40 ml. of 1 per cent bromthymol blue stock solution into a 1000-ml. volumetric flask and make up almost to mark, using CO_2-free distilled water. Mix thoroughly and remove a drop on an artists' spot plate. Compare color with bromthymol blue color chart. Adjust indicator to pH 6.8, using very dilute NaOH solution (about 0.05 N) if indicator is too acid, or dilute HNO_3 solution (about 0.05 N) if it is too alkaline, mixing thoroughly and sampling after each addition.

Bromcresol Green pH Indicator:

Prepare in a similar manner, using 40 ml. of 1 per cent bromcresol green stock per liter and adjusting pH to 4.6.

Chlorphenol Red pH Indicator:

Use 40 ml. of 1 per cent chlorphenol red stock per liter and adjust pH to 5.8.

Phenol Red pH Indicator:

Use 20 ml. of 1 per cent phenol red stock per liter and adjust pH to 7.4.

Hydrochloric Acid:

Chemically pure concentrated acid (free from arsenic and phosphates).

Nitric Acid:

Chemically pure concentrated acid (free from arsenic and phosphates).

N/2 Hydrochloric Acid Solution:

Forty ml. concentrated hydrochloric acid diluted to 1 liter with distilled water.

Potassium Sulfate:

Chemically pure powder, low in nitrogen content.

R.C.M. Color Standards

The preparation of these standards from organic dyes is an elaborate and involved process. Details of preparation are to follow but it may be stated that the various standards are being replaced as rapidly as research makes it possible by solutions of inorganic ions in media of approximately identical physical characteristics to the test solutions formed in the colorimetric analyses.

Soil Phosphate:

The stock reagents are prepared as follows:

Reagent "A"—Brilliant Wool Blue G. Extra, Schultz, No. 565.
Dissolve 0.5 gram in 1000 ml. distilled water.

Reagent "B"—Metanil Yellow, Schultz No. 134.
Dissolve 0.25 gram in 1000 ml. distilled water.

Reagent "C"—Erythrosine, Schultz No. 592.
Dissolve 0.1 gram in 500 ml. distilled water.

Reagent "D"—Brilliant Milling Green B, Schultz No. 503.
Dissolve 0.5 gram in 1000 ml. distilled water.

To prepare the color standards, mix the following portions in the order given, mixing and washing down the side of the flask with sufficient distilled water after each addition. Start with about 150 ml. of distilled water in each flask. The stock reagents and standards should be stored in a dark place away from sunlight.

P_2O_5 Group	ml. of "A"	ml. of "B"	ml. of "C"	ml. of "D"	ml. Total Volume
		Standard Series "X"			
High	33	11	0	0	250
Medium	25	10	0	0	250
Doubtful	14	6	0	0	250
Low	Water				250
		Standard Series "Y"			
High	30	9	0.5	2.5	250
Medium	17	4.5	1.0	0.2	250
Doubtful	6	2.0	0.5	2.0	250
Low	0	2.5	0	0	250

High-Register Soil Phosphate:

The stock reagents are prepared from:

Reagent 2A—Brilliant Wool Blue G. Extra, Schultz No. 565.
Dissolve 1.0 gram in 1000 ml. distilled water.

Reagents B, C and D—These reagents are prepared in a similar manner to those for the soil phosphate standards.

In preparing the standards, the following portions are mixed in the order given with subsequent additions of water:

No.	Per Cent P_2O_5	Lbs./a-ft.	Equiv. to No. ml. of 0.1 mg. P_2O_5/ml. made up to 100 ml.	ml. "2A"	ml. "B"	ml. "C"	ml. "D"	ml. Total Volume
I	.004	100	8	15	9	0.5	2.5	250
II	.006	150	12	21	10	0.5	5.0	250
III	.008	200	16	28	11	0.5	7.0	250
IV	.010	250	20	35	13	0.5	10.0	250
V	.012	300	24	45	15	0.5	18.0	250
VI	.014	350	28	55	17	1.0	25.0	250
VII	.016	400	32	65	20	1.0	35.0	250

Phosphate in Cane Juice Color Standards:

The stock reagents (reagents A, B, C and D) are identical to those used for soil phosphate standards. The color standards are prepared as follows:

No.	Equiv. to No. ml. of 0.1 mg. P_2O_5/ml. made up to 100 ml.	ml. "A"	ml. "B"	ml. "C"	ml. "D"	ml. Total Volume
1	4	13	4.3	0.5	0.3	250
2	6	18	5.0	0.5	1.0	250
3	8	30	9.0	0.5	2.5	250
4	10	35	9.0	0.5	4.0	250
5	12	42	10.0	0.5	5.0	250
6	14	48	10.0	0.5	6.0	250
7	16	55	11.0	0.5	7.0	250
8	18	62	12.0	0.5	8.0	250

Soil Phosphate Fixation Color Standards:

The stock solutions are made up as follows:

A. Methylene Blue—Merck Reagent Grade: Transfer 0.50 gm. methylene blue to a 500-ml. volumetric flask; add sufficient distilled water to dissolve the dye. Make up to volume, stopper and mix solutions thoroughly. Use this as stock solution "A."

B. Aniline Yellow—Eimer & Amend Tartrazine: Transfer 0.25 gm. aniline yellow to a 250-ml. volumetric flask. Dissolve the dye with sufficient water and make up to mark. Stopper and mix solution thoroughly. Filter. Use filtrate as stock solution "B."

C. Fuschsin: Transfer 0.10 gm. fuschsin to a 500-ml. volumetric flask. Add sufficient hot water (below boiling point) to dissolve the crystals. Cool to room temperature and make up to mark. Stopper and mix solution thoroughly. Use this as stock solution "C."

To 250-ml. volumetric flasks pipette the given amounts of reagents "A," "B" and "C" as specified in the table, subject to the following instructions:

1. To each flask add 175 ml. distilled water.

2. Pipette accurately the required amount of methylene blue stock solution "A" to each flask. Wash down reagent adhering to neck of flask. Mix the liquid thoroughly.

3. Add by pipetting the required amount of aniline yellow, stock solution "B" and repeat the process of washing and mixing.

4. Add by pipetting the required amount of fuschsin, stock solution "C" and repeat the washing and mixing process.

5. Make up to mark with distilled water. Stopper and mix the solution thoroughly.

6. Remove the stopper, add 1 drop thymol-menthol solution to each 250-ml. color standard as prepared above. Stopper and mix thoroughly. The color standards are now ready to be transferred to vials. (Always mix contents thoroughly

before transferring.) To prevent contamination by organic substances present in corks, the vials should be rubber stoppered.

7. The above order of adding reagents should be strictly adhered to in order to insure complete blending of colors and to prevent precipitation. The oily bactericide should be added after the solution has been made up to volume and mixed. It has been found that the oil will absorb a very small amount of red coloring matter if it is allowed to come into contact with the concentrated reagents.

Index No.	ml. Methylene Blue "A"	ml. Aniline Yellow "B"	ml. Fuschsin "C"	ml. Total Volume
0	25.0	4.00	4.00	250
10	19.50	3.00	3.00	250
20	14.50	2.00	3.00	250
30	10.50	1.60	3.40	250
40	7.50	1.00	2.60	250
50	5.00	0.70	2.50	250
60	3.50	0.50	2.10	250
70	2.20	0.40	1.60	250
80	2.50	0.50	1.50	500
90	1.60	0.30	0.80	500

Soil Nitrogen Color Standards:

The stock solutions to be used in the preparation of these color standards are made up as follows:

Solution III.—Dissolve 0.1 gm. Erythrosine 592 in a liter of distilled water.

Solution IV.—Dissolve 0.5 gm. Metanil Yellow in a liter of distilled water.

The following proportions of Solutions III and IV are used to prepare the ammoniacal nitrogen color standards:

(1 cup, 10 gm. soil—50 ml. N—5 ml. filtrate)

No.	Per Cent N (NH_4)	Lbs. N/ a-ft.	0.5 gm./L ml. M. Yel.	0.1 gm./L ml. Eryth. (red)	ml. Total Volume
1	.0002	5	1.00	0.50	250
2	.0006	15	2.20	0.90	250
3	.0010	25	3.60	1.70	250
4	.0014	35	4.70	2.70	250
5	.0018	45	6.50	4.00	250
6	.0024	60	9.00	6.00	250
7	.0030	75	12.00	9.00	250
8	.0040	100	18.00	15.00	250

The nitrate nitrogen color standards consist of mixtures of aqueous solutions of potassium chromate and potassium dichromate. Each solution contains 5 grams of the salt per liter. The exact proportion in which the solutions must be mixed to give each standard is presented in the following table:

Solution I—$K_2Cr_2O_7$ 5 gm./L
Solution II—K_2CrO_4 5 gm./L

Standard equiv. to Lbs. NO_3 N/a-ft.	ml. Soln. I per 250 ml.	ml. Soln. II per 250 ml.
5	3.25	0
13	7.50	0
25	7.50	7.50
38	8.75	13.75
50	11.25	21.25
75	13.75	31.25
100	16.25	43.75
125	20.00	80.00

Solutions I and II in the proportions given above are added to a 250-ml. volumetric flask from burettes and made up to volume with distilled water. The solution is mixed and transferred into vials which are then stoppered and sealed.

Detailed R.C.M. Procedures

Detailed directions for analyses of soil, plant material, crusher juice, mill by-products, and irrigation waters follow.

Rapid Estimation of Available Nitrogen In Soils

Equipment Required

6 Flasks, Erlenmeyer, 125-ml. cap.
12 Beakers, Pyrex, 100-ml. cap.
1 Box No. 12 Whatman folded filter paper, 15 cm.
1 Funnel support.
6 Funnels, 65 mm.
1 Pipette, Exax, volumetric, 25-ml. cap.
1 Pipette, Exax, volumetric, 5-ml. cap.
1 Metal soil cup, 10-gram cap.
1 Spatula, stainless steel, 4-inch blade.
1 Support, iron, 6 in. by 9 in.
1 Clamp, burette, castaloy, large, with rubber-covered jaws.
1 Burette, dispensing, 250-ml. cap.
1 Cover for 250-ml. dispensing burette.
10 Vials, shell, tall form.
10 Vials, tall form (reserved for ammoniacal and Total N).
1 Pipette, special, to deliver Reagent 7, N, 2-ml. cap.
1 Pipette, special, to deliver Reagent 6, N, 1-ml. cap.
10 Rubber stoppers, No. 00.
1 Electric hot plate.
1 Set ammonia color standards
1 Set nitrate color standards
} in box with 2 pcs. of black sateen.
6 Glass rods, 4 inches long.
1 Phosphate illuminator.
1 Bottle, dropping, pipette stopper with nipple, 30-ml. cap. for Reagent 6A, N.
1 Cylinder, graduated, 25-ml. cap.

1 Pipette, Mohr, 5-ml. cap.
1 Pipette, Mohr, 10-ml. cap.
1 gal. Reagent 5, N.
¼ pt. Reagent 6, N.
¼ pt. Reagent 6A, N.
¼ pt. Reagent 7, N in g. s. b.
1 qt. Reagent 8, N.
1 qt. Reagent 9, N.
1 qt. Reagent 18, Total N.

Additional Equipment Recommended

2 Burettes, dispensing, 250-ml. cap. (with covers) instead of 1 cylinder, graduated, 25-ml. cap. (with proper supports).

Equipment Required For Soil Nitrogen Determinations Not Included in Potash or Phosphate Ensembles

10 Vials, shell, tall form (reserved for ammoniacal and Total N).
12 Beakers, Pyrex, 100-ml. cap.
1 Box No. 12 Whatman folded filter paper, 15 cm.
1 Pipette, Exax, volumetric, 25-ml. cap.
1 Pipette, Exax, volumetric, 5-ml. cap.
1 Pipette, special, to deliver Reagent 7, N, 2-ml. cap.
1 Pipette, special, to deliver Reagant 6, N, 1-ml. cap.
10 Rubber stoppers, No. 00.
1 Set ammonia color standards } in box with 2 pcs. of black sateen.
1 Set nitrate color standards }
6 Glass rods, 4 inches long.
1 Bottle, dropping, 30-ml. cap., for Reagent 6A, N.
1 Cylinder, graduated, 25-ml. cap.
1 Pipette, Mohr, 5-ml. cap.
1 Pipette, Mohl, 10-ml. cap.
1 gal. Reagent 5, N.
¼ pt. Reagent 6, N.
¼ pt. Reagent 6A, N.
¼ pt. Reagent 7, N g. s. b.
1 qt. Reagent 8, N.
1 qt. Reagent 9, N.
1 qt. Reagent 18, Total N.

Extraction

1. Fill a 10-gram metal cup with prepared soil and level off with a stainless steel spatula. Transfer to a 125-ml. Erlenmeyer flask.
2. Add 50 ml. of Reagent 5, N, from a 250-ml. dispensing burette.
3. Shake 1 minute.
4. Filter through No. 12 Whatman folded filter paper, 15 cm., into a 100-ml. beaker.

Determination of Ammoniacal Nitrogen

1. Pipette 5 ml. of the filtrate (step No. 4 above) into a tall form, shell vial.

Precautions: Reserve a set of these vials exclusively for ammoniacal and total nitrogen determinations. Do not use vials employed in other studies. Do not carry on ammoniacal nitrogen determinations until all bottles containing Reagent 9, N, are closed and all vessels containing this reagent have been emptied and washed.

2. Add 4 drops Reagent 6A, N.

3. Add 1 ml. Reagent 6, N, with the special pipette.

4. Stopper vial with a No. 00 rubber stopper and let stand for 1 minute.

5. Shake vigorously to disperse all the precipitate that may be formed upon the addition of Reagent 6, N, to the filtrate.

6. Transfer the test vial to the rack of ammonia color standards, place on a phosphate illuminator and make comparisons.

7. Results are directly indicated on the standard color vials and are expressed as per cent N (ammoniacal) and pounds N per acre-foot of 2.5 million pounds soil.

8. When the amount of ammoniacal nitrogen exceeds 100 pounds per acre-foot, proceed as follows:

9. Using a 5-ml. Mohr pipette, transfer a suitable aliquot (2½, 2, 1, ½ ml., etc.) of the original 10-gram-50-ml. extract into a tall vial.

10. Dilute to 5 ml. with Reagent 18, Total N by means of a 10-ml. Mohr pipette.

Precaution: In making dilutions with Reagent 18, Total N, pour out enough into a beaker and use the solution in the beaker. After completion of a set of analyses, discard the remaining solution.

11. Proceed with steps Nos. 2 to 6 of ammoniacal nitrogen.

12. Multiply the results by a factor obtained by dividing 5 by the aliquot of filtrate taken. For example:

Aliquot taken = $2\frac{1}{2}$ ml. — Multiply result by $5/2\frac{1}{2}$ or 2.
Aliquot taken = 2 ml. — Multiply result by 5/2 or $2\frac{1}{2}$.
Aliquot taken = $\frac{1}{2}$ ml. — Multiply result by $5/\frac{1}{2}$ or 10.

Determination of Nitrate Nitrogen

1. Pipette 25 ml. of filtrate (step No. 4, extraction) into a clean 100-ml. beaker.

2. Evaporate on a hot plate nearly to dryness at low heat. (The residue may be dry but should *not* be allowed to bake.)

Precaution: It is not desirable to proceed beyond step No. 5, below, while other nitrogen studies (ammoniacal and total nitrogen) are in progress. It will be convenient to postpone steps Nos. 3 to 10 until other nitrogen studies have been terminated.

3. Let cool 1 minute and add 2 ml. of Reagent 7, N, using the special pipette.

4. Mix well by means of the glass rod, breaking up the residue to insure its intimate contact with the reagent. Let stand 5 minutes.

5. Add, either from a graduated cylinder or a 250-ml. dispensing burette, 10 ml. of Reagent 8, N, and stir well to effect solution.

6. Using the same graduate as in step No. 5, or a separate 250-ml. dispensing

burette, add 15 ml. of Reagent 9, N, agitating vigorously the contents of the beaker at the same time.

Precaution: Keep bottles of Reagent 9, N, in a separate place away from other reagents. When through dispensing this reagent, empty graduates or burettes and wash immediately. Empty and wash other vessels to which Reagent 9, N, has been added as soon as convenient.

7. Allow any insoluble residue to settle and decant the clear portion of the liquid into a tall form vial.

8. Transfer the test vial to the rack of nitrate standards placed on the phosphate illuminator and make comparisons.

9. Read off directly from the standard vial the concentration of nitrogen expressed as per cent N (nitrate) and pounds of N per acre-foot.

10. When the amount of nitrate nitrogen in the soil exceeds 125 pounds per acre-foot, proceed as follows:

11. Using a 10-ml. Mohr pipette, transfer a suitable aliquot (4 ml., 2 ml., 1 ml., etc.) of the solution in which the nitrate color has been developed (step No. 6 above) into a tall vial. Wash the pipette.

12. Add with the 10-ml. Mohr pipette amounts of Reagent 8, N, indicated in the tabulation following step No. 14, below.

Precaution: Pour out enough Reagent 8, N, into a beaker and use the solution in the beaker, discarding the remaining solution after completion of a set of analyses.

13. Cover vial with index finger and mix solution completely by inverting several times.

14. Compare with standards and multiply results by a factor obtained by dividing the total volume by the aliquot taken:

Aliquot Taken	Reagent 8, N, Added	Total Volume	Factor
4	4	8	2
2	4	6	3
2	6	8	4
1	4	5	5

Precautions

1. Keep Reagent 9, N, when not in actual use, in a separate place away from other reagents. Do not use when determination of nitrogen in forms other than the nitrate are in progress.

2. Color standards should be kept in closed boxes when not in actual use.

3. Reagents 5, N, and 7, N, should not be put in the same cupboard with or left adjacent to concentrated nitric acid or Reagent 2, K_2O.

Rapid Estimation of Total Nitrogen In Filter Cake

(Sample to be weighed out on an analytical balance)

1. Obtain a representative sample of the filter cake.

2. Break up large lumps by crumbling with hand. Mix material thoroughly. The sample is now ready for analysis without drying.

3. Weigh 2.5 gm. of the sample and transfer to Kjeldahl flask, R. C. M. Drop in 4 glass beads. Add 1 to 2 grams of special potassium sulfate. Introduce 20 ml. Reagent 15, Total N, with graduated cylinder. Let stand 5 minutes.

4. Place Kjeldahl flask on heater to digest. Insert gas absorption Hengar tube into neck of flask.

5. Digest for *one* hour.

6. Cool to room temperature. Add 100 ml. Reagent 8, N, from graduated cylinder, washing down neck of Kjeldahl flask. Cool slightly. Transfer by pouring into 250-ml. volumetric flask through a 65-mm. funnel. Rinse Kjeldahl flask several times with Reagent 8, N, transferring the rinsings to the 250-ml. volumetric flask. When the solution in the volumetric flask is cool, make volume up to 250-ml. mark with Reagent 8, N. Stopper and mix thoroughly.

7. Pipette 10 ml. of the solution from the volumetric flask into a clean Kjeldahl flask. Drop in 2 glass beads. Add 100 ml. Reagent 8, N, and 20 ml. Reagent 17, Total N. Connect flask with distillation apparatus. Shake flask slightly. Immediately place Kjeldahl flask on heater and introduce distillation outlet into the calibrated test tube to which has been added 15 ml. Reagent 16, Total N. The test tube is immersed in cold water in a 500-ml. Erlenmeyer flask which acts as a condenser.

8. Remove test tube when nearly 50 ml. have been distilled over. Cool distillate to room temperature. (Tube is removed before heat is shut off from the distilling flask.)

9. Make volume to exactly 50 ml. with Reagent 8, N. Mix thoroughly.

10. Pipette 5 ml. into comparison vial. Add 1 ml. Reagent 6, N; let stand 3 minutes. Stopper and mix well. (The original distillate may be used or dilutions of the original may be made. However, for both original distillate or diluted aliquots, 5-ml. portions are used for color development. The dilution should be thoroughly mixed before the 5-ml. portions are withdrawn for color development with Reagent 6, N.)

11. Compare with *Soil Ammoniacal Standards,* using the illuminator. Refer to table for results.

Data for Total N in Filter Cake

2.5-gm. Sample — 250 ml. Digested Soln.
10-ml. aliquot used in distillation, equivalent to 0.1-gm. sample.
50-ml. Distillate Obtained.

PER CENT TOTAL N IN FILTER CAKE

Reading Tube No.	5 ml. Orig. Distillate		Distillate diluted as indicated below:* 5 ml. Orig. Dist. 5 ml. Reag. 8		5 ml. Orig. Dist. 15 ml. Reag. 8		5 ml. Orig. Dist. 35 ml. Reag. 8	
	Per Cent N	lb/ton	Per Cent N	lb/ton	Per Cent N	lb/ton	Per Cent N	lb/ton
1	..	..	..	..	..	..	..	..
2	.06	1.2	.12	2.4	..	..	..	..
3	.10	2.0	.20	4.0	.40	8.0	.80	16.0
4	.14	2.8	.28	5.6	.56	11.2	1.12	22.4
5	.18	3.6	.36	7.2	.72	14.4	1.44	28.8
6	.24	4.8	.48	9.6	.96	19.2	1.92	39.4
7	.30	6.0	.60	12.0	1.20	24.0	2.40	48.0
8	.40	8.0	.80	16.0	1.60	32.0	3.20	64.0

* Use 5 ml. of the final solution, diluted in proportions as indicated, for color development.

Rapid Estimation of Total Nitrogen in Soil

1. Quarter prepared soil until about one-half pound remains.

2. Grind this material to a fine powder with a mortar and pestle. Mix thoroughly.

3. Weigh 2.5 grams of the sample and transfer to a 300-ml. Kjeldahl flask. Drop 4 glass beads into the flask. Add 1 to 2 grams of potassium sulfate. Introduce 20 ml. Reagent 15, Total N, from a graduated cylinder.

4. Let stand for about 5 minutes. Fit a Hengar tube into the neck of the Kjeldahl flask and place it on the electric heater to digest.

5. Digest for 1 hour. Using the wooden clamp, remove flask to the box. Cool to room temperature.

6. Add 100 ml. Reagent 18, Total N, from a graduated cylinder, washing down the neck of the flask. Cool. Carefully decant off the supernatent liquid into a 250-ml. volumetric flask through a 65-mm. funnel. Rinse the Kjeldahl flask several times with small portions of Reagent 18, Total N, letting sediment settle and decanting off supernatent liquid each time into the volumetric flask. When the solution in the volumetric flask is cool, make volume up to 250-ml. mark with Reagent 18, Total N. Stopper and mix thoroughly.

7. To a large, calibrated test tube add 15 ml. of Reagent 16, Total N. Immerse it in cold water in a 500-ml. Erlenmeyer flask which acts as a condenser. Set the condenser on an iron support shelf and arrange this unit adjacent to heater unit for the distillation.

8. Pipette 10 ml. of the solution from the volumetric flask into a clean Kjeldahl flask. Drop in 2 glass beads. Add 100 ml. Reagent 18, Total N, and a pinch of granulated zinc (20 mesh). Turn on heater current. Add 20 ml. Reagent 17, Total N, from a special dispensing burette, introducing it carefully down the inner side of the flask. Immediately attach the connecting bulb assembly, stoppering "trap end" tightly to the Kjeldahl flask. The outlet tube is put into the test tube with the tip momentarily withheld above the level of Reagent 16, Total N, while the contents of the Kjeldahl flask is agitated thoroughly. After mixing, the tip of the distillation outlet tube is immersed in the 15 ml. of Reagent 16, Total N. Immediately place the Kjeldahl flask on the heater.

9. Distillation is continued until the distillate reaches the level marked on the test tube, when the condensing flask and tube are immediately removed from the distillation outlet. The heater may then be turned off and the connecting bulk assembly removed. The test tube is removed from the Erlenmeyer flask and placed on the tube rack to cool.

10. Make volume up to exactly 50 ml. with Reagent 18, Total N. Insert a clean rubber stopper and mix thoroughly.

11. Pipette 5 ml. into a comparison vial. Add 1 ml. Reagent 6, N; let stand 3 minutes. Stopper and mix well. (The original distillate may be used or dilutions of the original may be made. However, for both original distillate or diluted aliquots, 5-ml. portions are used for color development. The dilution should be thoroughly mixed before the 5-ml. portions are withdrawn for color development with Reagent 6, N.)

12. Compare with *Soil Ammoniacal Standards,* using the illuminator. Refer to table for results.

DATA FOR TOTAL N IN SOILS

2.5-gm. sample — 250 ml. digested soln.
10-ml. aliquot used in distillation equiv. to 0.1-gm. sample.
50 ml. distillate obtained.

Reading Tube No.	5 ml. Original Distillate		5 ml. Original Dist. 5 ml. Reag. 18, T N		5 ml. Original Dist. 15 ml. Reag. 18, T N	
	Per Cent N	lb/a-ft.	Per Cent N	lb/a-ft.	Per Cent N	lb/a-ft.
2	.06	1,500	.12	3,000	..	.....
3	.10	2,500	.20	5,000	.40	10,000
4	.14	3,500	.28	7,000	.56	14,000
5	.18	4,500	.36	9,000	.72	18,000
6	.24	6,000	.48	12,000	.96	24,000
7	.30	7,500	.60	15,000	1.20	30,000
8	.40	10,000	.80	20,000	1.60	40,000

Rapid Determination of Nitrogen in Mill Water and in Water Containing Organic Matter

Use the same equipment as is required in making determinations of total nitrogen in cane juice and available nitrogen in soil.

General Procedure:

Collect 1 gallon of water as representative of the sample to be analyzed. Thoroughly mix in bottle. Filter about 250 ml. through a Whatman No. 12 15-cm. filter paper. Nitrogen is determined by two procedures: (I) Organic plus ammoniacal nitrogen determination and (II) nitrate nitrogen analysis.

I. Determination of Organic Plus Ammoniacal Nitrogen:

1. Measure 100 ml. of the filtered sample and transfer to a Kjeldahl flask. Drop 2 glass beads into flask. Add approximately .3 gm. potassium sulfate powder (about 1/3 spoonful—small horn spoon supplied in N-in-cane-juice assembly). Introduce 4 ml. Reagent 15, Total Nitrogen, by special pipette.

2. Heater is turned on and the Kjeldahl flask is placed on it.

3. Boil until water is evaporated and white fumes are formed in neck of flask. Attach Hengar tube and continue digestion for 15 minutes. Total time (from moment flask is set on heater to completion of digestion) required is about 1 hour.

4. Remove flask from heater and cool to room temperature. When cool, add 100 ml. Reagent 18, Total Nitrogen, from graduated cylinder, washing down neck of Kjeldahl flask. Let stand to cool.

5. Pipette 15 ml. Reagent 16, Total Nitrogen, into a calibrated test tube and immerse test tube in a 500 ml. wide-mouth Erlenmeyer flask containing about ¾ tap water. Set flask and tube on iron support shelf. Arrange this unit and heater unit for the distillation.

6. Add 20 ml. Reagent 17, Total Nitrogen, to the Kjeldahl flask. Attach the

connecting bulb assembly, stoppering "trap-end" tightly into the flask. The outlet tube is put into the test tube with the tip momentarily withheld above the level of the Reagent 16, Total Nitrogen, while the mixture in the Kjeldahl flask is stirred thoroughly. After mixing, the tip of the distillation outlet tube is immersed in the 15 ml. of Reagent 16, Total Nitrogen. The flask is placed on the heater with the current connected.

7. Distillation is continued until the distillate reaches the level marked on the test tube, whence the water flask and tube are immediately removed from the distillation outlet. The heat may then be turned off and the connecting bulb assembly removed.

8. When the distillate has cooled to room temperature the volume is made exactly to 50 ml. with Reagent 18, Total Nitrogen. The solution is mixed thoroughly.

9. Pipette 5 ml. aliquots into each of 2 comparison vials. Add 1 ml. Reagent 6, Nitrogen, into each tube. Let stand 1 minute. Stopper and mix well.

10. Compare with *Soil Ammoniacal Standards*, using the illuminator. Refer to the table for results.

11. When the result is indicated to be above that shown in the first column of data, diluted solutions may be made as indicated on the chart. Five ml. of diluted solution are taken for analysis.

Results obtained from the above test include organic and ammoniacal nitrogen.

TABLE FOR N IN MILL WATER (ORGANIC AND AMMONIACAL N)

Parts per million (p.p.m.) N and pounds per million gallons (lb/mil. gal.)

Tube No.	5 ml. Orig. Distillate		10 ml. Orig. Distillate 10 ml. Reagent 18, T N Take 5 ml. Aliquots		10 ml. Orig. Distillate 30 ml. Reagent 18, T N Take 5 ml. Aliquots	
	p.p.m.	lb/mil. gal.	p.p.m.	lb/mil. gal.	p.p.m.	lb/mil. gal.
1	..	...	..	...	..	...
2	0.5	4.2	1.1	9.1	2.4	19.9
3	0.9	7.5	1.9	15.8	4.0	33.2
4	1.3	10.8	2.7	22.4	5.6	46.5
5	1.7	14.1	3.5	29.0	7.2	59.8
6	2.3	19.1	4.7	39.0	9.6	79.7
7	2.9	24.1	5.9	49.0	12.0	99.6
8	3.9	32.4	7.9	65.6	16.0	132.8

II. Nitrate Nitrogen:

To determine nitrate nitrogen, use the nitrate nitrogen in irrigation water method.

RAPID DETERMINATION OF NITROGEN IN IRRIGATION WATERS

(Water Other Than Mill Waters or Waters Containing Organic Matter)

Use the same equipment as is required in making determinations of available nitrogen in soils.

General Procedure:

Collect 1 gallon of water. Mix thoroughly and filter sufficient amount for analysis (approximately 250 ml.). Use Whatman No. 12, 15-cm. filter paper.

I. To Determine Ammoniacal Nitrogen in Irrigation Water:

1. Pipette 5 ml. filtered sample into each of 2 test vials.
2. Add 1 ml. Reagent 6, Nitrogen. Let stand 1 minute. Stopper and mix well.
3. Compare with *Soil Ammoniacal Nitrogen Standards,* using the illuminator. Refer to table for results.

AMMONIACAL NITROGEN IN IRRIGATION WATER

Std. No.	p.p.m. N	lb N/mil. gal.
1	0.4	3
2	1.2	10
3	2.0	17
4	2.8	23
5	3.6	30
6	4.8	40
7	6.0	50
8	8.0	67

II. To Determine Nitrate Nitrogen in Irrigation Water:

1. Pipette 25 ml. of the filtered water into a 100-ml. beaker and evaporate nearly to dryness on hot plate at low heat.
2. When cool, add 2 ml. Reagent 7, Nitrogen, using special pipette.
3. Mix well by means of glass rod, breaking up the residue to insure its intimate contact with the reagent. Let stand 5 minutes.
4. Add from a graduated cylinder 10 ml. Reagent 8, Nitrogen, and stir well to effect solution.
5. Add 15 ml. Reagent 9, Nitrogen, stirring the solution in the beaker while Reagent 9, Nitrogen, is being added. (Add by using 25 ml. graduated cylinder or dispensing burette.) Let final solution cool.
6. Decant liquid into 2 test vials. Compare with *Soil Nitrate Nitrogen Standards,* using the illuminator. The color tubes are designated as 1 to 8, beginning with the lowest standard. Refer to table for results.

NITRATE NITROGEN IN IRRIGATION WATER

Std. No.	p.p.m. N	lb N/mil. gal.
1	0.4	3
2	1.0	8
3	2.0	17
4	3.0	25
5	4.0	33
6	6.0	50
7	8.0	67
8	10.0	83

Rapid Estimation of Nitrogen in Cane Juice

Equipment Required

4 Supports, iron, 6 in. x 9 in., with rods 15 in.
2 Heaters, precision, electric.

2 Clamps, burette, castaloy, large, with asbestos-covered jaws.
2 Support shelves, round.
6 Flasks, Kjeldahl, 300-ml. cap.
1 Box for Kjeldahl flasks.
1 Rack, test tube.
4 Flasks, Erlenmeyer, wide mouth, 500 ml.
6 Test tubes, Pyrex, calib. 50 ml., 200 x 29 mm.
6 Rubber stoppers, No. 6.
2 Connecting bulb assemblies.
2 Hengar tubes, micro-size, with filter paper tape.
1 Pipette (bacteriological), 1-ml. cap.
1 Pipette, special, for Reagent 15, Total N.
1 Bottle (g. s. b.), 6 oz.
1 Pipette, volumetric, 5-ml. cap.*
1 Pipette, special, to deliver Reagent 6, N, 1-ml. cap.*
1 Burette, special, dispensing, with rubber tube and pinchcock for delivering Reagent 17, Total N.
1 Cylinder, graduated, 100-ml. cap.
2 Juice screens.
1 Clamp, wooden.
1 Set ammonia nitrogen in soil color standards.*
1 Vial block.*
1 Phosphate illuminator.*
10 Rubber stoppers, No. 00.*
12 Shell vials, tall form.*
2 oz. Glass beads, perforated.
1 Small horn spoon.
1 Pipette, volumetric, 15-ml. cap.
2 Pyrex beakers, 600-ml. cap.
6 Pyrex beakers, 400-ml. cap.
6 Pyrex beakers, 250-ml. cap.
1 Pipette, volumetric, 25-ml. cap.
¼ lb. Potassium sulfate C. P. Powd.
½ pt. Reagent 15, Total N.
1 qt. Reagent 17, Total N.
1 qt. Reagent 16, Total N.
1 gal. Reagent 18, Total N.*
¼ pt. Reagent 6, N.*
¼ pt. Cane juice preservative.

* Note: Standard equipment included in soil phosphate and available soil nitrogen assemblies.

Preliminary Procedure

1. Use fresh, untreated cane juice or juice treated with the proper preservative supplied for rapid chemical methods.

2. Obtain a representative sample. Mix well. Let stand for exactly 15

minutes and then decant about 250 to 500 ml. into a clean 400- or 600-ml. beaker. Pass decanted juice through a juice screen into a 250- or 400-ml. beaker.

3. Immediately before pipetting portions for analysis, mix the screened juice thoroughly.

Determination of Total Nitrogen

1. (a) Pipette 1 ml. of screened juice into a 300-ml. Kjeldahl flask, taking care to deliver the juice to the bottom of the flask.

(b) Drop 2 glass beads into the flask.

(c) Add 1/3 of a small horn spoonful of potassium sulfate (K_2SO_4) powder.

(d) Introduce 4 ml. of Reagent 15, Total N, with the special pipette.

2. Let stand for about 5 minutes. Fit a Hengar tube into the neck of the flask and place the Kjeldahl flask on the heater to digest.

3. Digest for ½ hour. Using the wooden clamp, remove flask to the box. Cool to room temperature.

4. Add 100 ml. of Reagent 18, Total N, from a graduated cylinder, washing down the neck of the Kjeldahl flask. Let stand to cool.

5. Add 15 ml. of Reagent 16, Total N, to a large calibrated test tube. Immerse in cold water in a 500-ml. Erlenmeyer flask. Set flask and tube on iron support shelf and arrange this unit adjacent to heater unit for the distillation.

6. With the solution in the Kjeldahl flask at room temperature, add 20 ml. Reagent 17, Total N, from the special dispensing burette. Turn on heater current. Immediately attach the connecting bulb assembly, stoppering "trap-end" tightly with the flask. The outlet tube is put into the test tube with the tip momentarily withheld above the level of Reagent 16, Total N, while the mixture in the Kjeldahl flask is stirred thoroughly. After mixing, the tip of the distillation outlet tube is immersed in the 15 ml. of Reagent 16, Total N. Place the Kjeldahl flask on the heater.

7. Distillation is continued until the distillate reaches the level marked on the test tube, when the water flask and tube are immediately removed from the distillation outlet. The heater may then be turned off and the connecting bulb assembly removed. The test tube is removed from the Erlenmeyer flask and placed on the tube rack to cool. Cool distillate to room temperature.

8. Make volume exactly up to the 50-ml. mark with Reagent 18, Total N. Stopper test tube with No. 6 rubber stopper and mix contents thoroughly by inverting several times.

9. Pipette 5 ml. into each of 2 comparison vials. Add 1 ml. Reagent 6, N, to each with the special pipette. Stopper and let stand 1 minute.

10. Compare on the illuminator with the ammonia nitrogen-in-soil color standards.

11. Refer to the table under column headed "5-ml. Aliquot" for result.

12. When the nitrogen content is as high as standard tube No. 7, pipette 25 ml. of the distillate into a clean, dry 250-ml. beaker, add 25 ml. Reagent 18, Total N, from a graduated cylinder and mix well. Proceed with steps Nos. 9 and 10. Refer to the table under the column headed "Diluted solution" for result.

PERCENTAGE TOTAL NITROGEN (N) IN CANE JUICES

5-ml. Aliquot		Diluted Solution	
Ammon. N. Std. Tube No.	Per Cent Total N in Juice	Ammon. N. Std. Tube No.	Per Cent Total N in Juice
1	Nil	1	...
2	.004	2	...
3	.008	3	...
4	.012	4	.026
5	.016	5	.034
6	.022	6	.046
7	.028	7	.058
8	.038	8	.078

Rapid Chemical Methods Nitrogen Studies

The following is an outline which may be employed as a guide in making nitrogen studies on the plantation.

The details included in the outline embrace:

I. Plan of experiment.
II. Collection of samples and preparation for analysis.
III. Analysis of plant material.

Note: Refer to directions for nitrogen in soil and nitrogen in juice for other analytical procedures.

(A modification and simplification of the rapid chemical method for determining nitrogen in plant material is now under study by L. E. Davis.)

I. Plan of Experiment:

Plan No. 1: A study by R. C. M. and field experiments of available nitrogen in the soil and of total nitrogen in the cane plant before there is millable cane, followed by total nitrogen in the juice of the millable cane.

1. Within the experimental area, select a sampling station which will include one plot of each treatment in the test. (If the layout is a "block arrangement" this sampling station may be any one "block" of plots.)

2. Within each of the plots in this sampling station, select a definite cane row (not an outside row) and secure a soil sample therefrom by compositing not less than 20 borings taken with a 2-inch auger to a depth of 12 inches about 12 inches away from the center of the cane row, on both sides of the center.

This soil sample should be taken as soon as the seed is planted or the ratoon crop is started, *before* any nitrogen fertilizer is applied. Determine the R. C. M. nitrogen in these soil samples (one for each treatment).

3. Just prior to each nitrogen application that is to be made to the experimental area, select (at random) 2 feet of line within the cane row that was originally sampled in each plot. Take a cane sample by cutting out *all* the cane growing on these 2 feet of line. Up to the time when the cane is about 4 or 5 months old, the entire plant

material itself may be prepared* and analyzed by R. C. M. for total nitrogen. When there is sufficient millable cane for a juice sample, the cane stalks may be topped, the millable cane crushed, and the crusher juice analyzed for its total nitrogen content. Thereafter, take a soil sample from this 2-foot section of row by compositing the soil from 6 borings, taken as for the original soil sample.

Analyze cane for total N, and soil sample for available N by R. C. M.

4. At harvest, analyze crushed juice of each plot in the sampling station for total nitrogen. Also take a soil sample as in the original sampling and analyze by R. C. M. for available nitrogen.

5. These data will be studied in connection with the results obtained in the experiment.

(*Note:* Always fill the auger holes that have been bored when taking out the soil sample.)

Plan No. 2: A study by R. C. M. of available soil nitrogen and total plant nitrogen at field sampling stations in crop cane.

1. Within any field of crop cane just planted or ratooned, choose a sampling station that will consist of twelve 50- to 60-foot rows of cane, and divide this station for 2 treatments (P. P. and X) into 4 sections as follows:

I 6 rows P. P.	III 6 rows X
II 6 rows X	IV 6 rows P. P.

2. Take 20 soil borings from the 2 center lines of each section and analyze the composited sample by R. C. M. for the amount of available nitrogen that is in the soil at the start of the crop.

3. Apply nitrogen as in the plantation practice to the 2 sections marked "P. P." Omit the nitrogen application on the 2 sections marked "X."

Watch for any definite evidence of slower growth, of any yellowing of leaves, etc., on the "X" plots and when this evidence is secured, take both a cane and a corresponding soil sample for nitrogen analysis from each of the 4 sections.

a. Take the cane sample by cutting out all cane growth from 2 feet of line within one of the 2 center rows; take its corresponding soil sample by making 6 borings with a 2-inch auger to a depth of 12 inches, at a distance of 12 inches from the center of this harvested row on both sides. Analyze both cane and soil for nitrogen content.

b. Definite evidence of slower growth may be secured by marking at least 20 shoots or stalks within the 2 center lines of each section and taking weekly growth measurements thereof.

5. Thereafter several procedures may be followed, depending on conditions:

(a) If no differences are apparent up to the time when the next nitrogen applica-

* See *II. Collection of Samples and Preparation for Analysis.*

tion is scheduled, the nitrogen can again be omitted from the "X" plots, etc., until differences do appear.

(b) If differences are apparent early, apply on the "X" plots the nitrogen that was omitted, and see if the cane can pick up its lost growth before the cane on the "P. P." plots is scheduled to get its next nitrogen application.

(c) If differences are not apparent until almost time for the next nitrogen application, make this next application of nitrogen the same to both the "P. P." and the "X" plots, and see if the cane on the "X" plots, which is minus one nitrogen application, can catch up with the cane on the "P. P." plots.

In all cases make determinations of the nitrogen in both the soil and plant whenever differences in cane growth and appearance are apparent.

This procedure should be followed for each scheduled application of nitrogen to the field, *a new sampling station being chosen each time.*

II. Collection of Samples and Preparation for Analysis:

(Obtaining a Specimen of Cane for the Determination of Nitrogen)

1. Depending upon the nature of the experiment or the purposes of the study, let it be assumed that a bundle of cane has been cut from a plot or definite area. The entire portion of the plant above ground is to be used for analysis. Obtain green weight, "A," of entire bundle.

2. Flatten out the bundle so that the individual stalks (with leaves attached) lie side by side, one layer deep. Select and reserve every fifth or sixth stalk starting at one end of the flattened bundle. If the sample is very small or the cane is quite young, then *all* of the collected cane may be reserved for analysis. (From ½ pound to 1 pound of *dry* sample will be sufficient.)

3. The selected cane and leaves should be finely chopped. Mix the chopped material thoroughly and quarter the sample down until but enough remains to fill a 1-pint Mason jar, after it has been ground and dried. Obtain weight, "B," of the reduced portion of finely chopped material. Spread out to dry in a warm, clean room. When dry, grind the sample to about the fineness of granulated sugar, using, if preferred, a grinder furnished by the Chemistry Department which is available with other supplies.

4. Place sample (in tared pan) in electric oven and dry at temperature between 80°C. – 100°C., preferably 80°C., until constant weight is obtained.

5. Allow pan containing dried sample to cool and obtain weight, "C," of oven-dry sample. When the sample has been dried to constant weight at the indicated temperatures, it is then assumed that the sample is moisture free. This will eliminate the determination of moisture for the calculation of results to moisture-free basis. Proceed with step No. 4, rapid determination of total N in plant material.

6. To calculate total nitrogen in the cane as it stood in the field:

(1) $\frac{A}{B} \times C = D$ Total moisture-free material in original bundle harvested

(2) D × per cent Total N = E (Amount in pounds or grams of total nitrogen in the cane as harvested, original bundle.)

Convert the value "E" by appropriate calculation into pounds nitrogen per acre or per line of known length.

III. Analysis of Plant Material:

(Rapid Determination of Total Nitrogen in Plant Material)

1. Obtain representative sample.
2. Chop green material and dry it to moisture-free condition.

(Consult "II. Collection of Samples and Preparation for Analysis" at this point.)

3. Grind dry sample, mix thoroughly and redry to moisture-free state.
4. Weigh accurately 2.5-gm. oven-dry sample and transfer to Kjeldahl flask, R. C. M. Drop in 4 glass beads. Add 1 to 2 grams of special potassium sulfate. Introduce 20 ml. Reagent 15, N, with graduated cylinder. Let stand 5 minutes.
5. Place Kjeldahl flask on heater to digest. Insert gas absorption Hengar tube into neck of flask.
6. Digest for *one* hour or until solution is clear and oxidation process is completed (very pale, straw color).
7. Cool to room temperature. Add 100 ml. Reagent 8, N, from graduated cylinder, washing down neck of Kjeldahl flask. Cool slightly. Transfer by pouring into 250-ml. volumetric flask through a 65-mm. funnel. Rinse Kjeldahl flask several times with Reagent 8, N, transferring the rinsings to the 250-ml. volumetric flask. When the solution in the volumetric flask is cool, make volume up to 250-ml. mark with Reagent 8, N. Stopper and mix thoroughly.
8. Pipette 10 ml. of the solution from the volumetric flask into a clean Kjeldahl flask. Drop in 2 glass beads. Add 100 ml. Reagent 8, N, and 20 ml. Reagent 17, N. Connect flask with distillation apparatus. Shake flask slightly. Immediately place Kjeldahl flask on heater and introduce distillation outlet into the calibrated test tube to which has been added 15 ml. Reagent 16, N. The test tube is immersed in cold water in a 500-ml. Erlenmeyer flask which acts as a condenser.
9. Remove test tube when nearly 50 ml. have been distilled over. Cool distillate to room temperature. (Tube is removed before heat is shut off from the distilling flask.)
10. Make volume to exactly 50 ml. with Reagent 8, N. Mix thoroughly.
11. Pipette 5 ml. into comparison vial. Add 1 ml. Reagent 6, N; let stand 1 minute. Stopper and mix well. (The original distillate may be used or dilutions of the original may be made. However, for both original distillate or diluted aliquots, 5-ml. portions are used for color development. The dilution should be thoroughly mixed before the 5-ml. portions are withdrawn for color development with Reagent 6, N.)
12. Compare with *Soil Ammoniacal Standards,* using the illuminator. Refer to table for results.

DATA FOR TOTAL N IN PLANT MATERIAL

2.5-gm. Sample — 250 ml. Digested Soln.
10-ml. aliquot used in distillation, equivalent to 0.1-gm. Sample.
50 ml. Distillate Obtained.

PER CENT TOTAL N IN PLANT MATERIAL, USING GROUND OVEN-DRY SAMPLE

Reading Tube No.	5 ml. Orig. Distillate	*Distillate Diluted as Indicated Below: 5 ml. Orig. Dist. 5 ml. Reag. 8	5 ml. Orig. Dist. 15 ml. Reag. 8	5 ml. Orig. Dist. 35 ml. Reag. 8
1	..	..	..	..
2	.06	.12	..	..
3	.10	.20	.40	.80
4	.14	.28	.56	1.12
5	.18	.36	.72	1.44
6	.24	.48	.96	1.92
7	.30	.60	1.20	2.40
8	.40	.80	1.60	3.20

* Use 5 ml. of the final solution, diluted in proportions as indicated, for color development.

Additional Equipment Required

Six only 250-ml. volumetric flasks, with rubber stoppers, pans for holding and drying sample, chopping knives, balance, oven and grinding machine.

RAPID ESTIMATION OF TOTAL NITROGEN IN MOLASSES

(Sample to be weighed out on an analytical balance)

Equipment Required

Items required for nitrogen-in-juice determinations.

1 Flask, volumetric, 100-ml. cap.

Procedure

1. Weigh out 5.0 grams of molasses in a weighing dish or small tared beaker.

2. Add about 25 ml. distilled water (N-free) and mix well by stirring.

3. Transfer to a 100-ml. volumetric flask, washing in with distilled water. Make volume up to the mark, stopper the flask and mix.

4. Proceed according to the directions for the rapid estimation of nitrogen in cane juices, steps Nos. 1-10, taking 1 ml. of the molasses solution *immediately* after mixing.

5. Refer to the table below under column headed "5-ml. Aliquot" for the result.

6. When the nitrogen content is as high as standard tube No. 7, pipette 25 ml. of the distillate into a clean, dry, 250-ml. beaker, add 25 ml. of distilled water (N-free) from a graduated cylinder and mix well. Proceed with steps Nos. 9 and 10, Rapid Estimation of Nitrogen in Cane Juices, and refer to the table below under the column headed "Diluted Solution" for the result.

TABLE FOR TOTAL NITROGEN IN MOLASSES

Ammon. N. Std. Tube No.	5-ml. Aliquot		Diluted Solution	
	Per Cent	lb/Ton	Per Cent	lb/Ton
2	.08	1.6	..	..
3	.16	3.2	..	..
4	.24	4.8	.52	10.4
5	.32	6.4	.68	13.6
6	.44	8.8	.92	18.4
7	.56	11.2	1.16	23.2
8	.76	15.2	1.56	31.2

Rapid Estimation of Phosphate in Soils

Equipment Required

1 Electric hot plate.
12 Flasks, Erlenmeyer, 125 ml.
12 Funnels, 65 mm.
1 Funnel support.
12 Beakers, Pyrex, 50-ml. cap.
24 Vials, shell, tall form.
1 pkg. No. 3 Munktell filter paper, 11 cm.
1 Pipette, volumetric, Exax, 10-ml. cap.
2 Burettes, dispensing, 250-ml. cap.
2 Covers for 250-ml. dispensing burettes.
2 Dropping bottles, with T. K. covers, 30-ml. cap.
1 Dropping bottle, pipette stopper with nipple, 30-ml. cap.
1 Metal soil cup, 10-gram cap.
1 Spatula, stainless steel, 4-inch blade.
1 Vial block.
1 Phosphate illuminator.
3 Supports, iron, 6 in. x 9 in.
1 Clamp, burette, Lincoln.
2 Clamps, burette, castaloy, large, with rubber-covered jaws.
1 Box containing phosphate color standards (X and Y and High-Register Standards).
1 gal. N/2 hydrochloric acid solution.
1 gal. Reagent 4, P_2O_5.
½ pt. Stannous chloride solution, in g. s. b.
½ lb. Conc. nitric acid, special, in g. s. b.
½ lb. Conc. hydrochloric acid, special, in g. s. b.

Procedure

1. Fill a 10-gram metal cup with prepared soil and level off with a stainless steel spatula. Transfer to a 125-ml. Erlenmeyer flask.

2. Add 30 ml. of N/2 hydrochloric acid solution from a 250-ml. dispensing burette.

3. Immediately swirl for ½ minute.

4. Immediately filter through No. 3 Munktell filter paper, 11 cm., into a 50-ml. beaker.

5. Transfer a 10-ml. portion of the filtrate with a pipette to another 50-ml. beaker.

6. Add 10 drops of conc. nitric acid from a dropping bottle with T. K. cover.

7. Evaporate to dryness on an electric hot plate at a low heat. *Do Not Bake.*

8. Add 10 drops of conc. nitric acid and 10 drops of conc. hydrochloric acid from dropping bottles with T. K. covers. Evaporate to dryness.

9. *Important:* If the residue is discolored, repeat the treatment with nitric acid and hydrochloric acid, followed by evaporation. If discoloration remains, repeat once more. If residue is still dark, disregard the discoloration and proceed.

10. Add 10 drops of conc. hydrochloric acid and evaporate to dryness. Repeat the addition of conc. hydrochloric acid and evaporation to dryness. *Do Not Bake.*

11. As soon as the residue is cool add 10 ml. of Reagent 4, P_2O_5, from a 250-ml. dispensing burette and swirl the contents of the beaker carefully for a few minutes to dissolve the residue. If the residue does not dissolve readily allow the mixture to stand for a while (with occasional swirling), but not longer than 15 minutes. If there is still a residue, disregard it and proceed.

12. Transfer to a phosphate vial, filling to about ¼ inch from the top. If there is a residue, transfer the supernatent liquid but not the residue.

13. Place the phosphate vial in a numbered vial block. Add stannous chloride, one drop at a time. Following each addition mix well, immediately place the vial in the phosphate illuminator and compare with the phosphate color standards, choosing that one of the 3 sets which best matches the hue and intensity of the solution in the vial. Continue until a maximum color intensity has been obtained.

14. Estimate the phosphate content of the soil by interpolation, referring to the attached chart and the table for high-register P_2O_5 standards. Record the data as "Low," "Doubtful," etc., as percentage of P_2O_5 and as pounds of P_2O_5 per acre-foot of soil. Note that each range (Low, Doubtful, etc.) includes the color standard tube so marked and all colors *more* intense up to, but not including, the next color standard. Colors in the high-register range are recorded as "High." For example:

"X" or "Y"	.0014 per cent	= Doubtful
	.0015 per cent	= Medium
	.0016 per cent	= Medium
High Register	.009 per cent	= High

15. Should the color developed be *equal to or greater* than High-Register P_2O_5 Standard VII, transfer ½ of the solution in the vial to another vial and add an equal volume of Reagent 4, P_2O_5.

16. Mix well and add 1 drop of stannous chloride solution. Compare with the high-register standards. Refer to the figures under "After Dilution" in the table and record as "High," per cent P_2O_5 and pounds P_2O_5 per acre-foot.

17. When the color is still darker than Standard VII, report the result as "High," greater than .032 per cent, greater than 800 pounds P_2O_5 per acre-foot, or

$>$.032 per cent $>$800 Lbs./a-ft.

Precautions

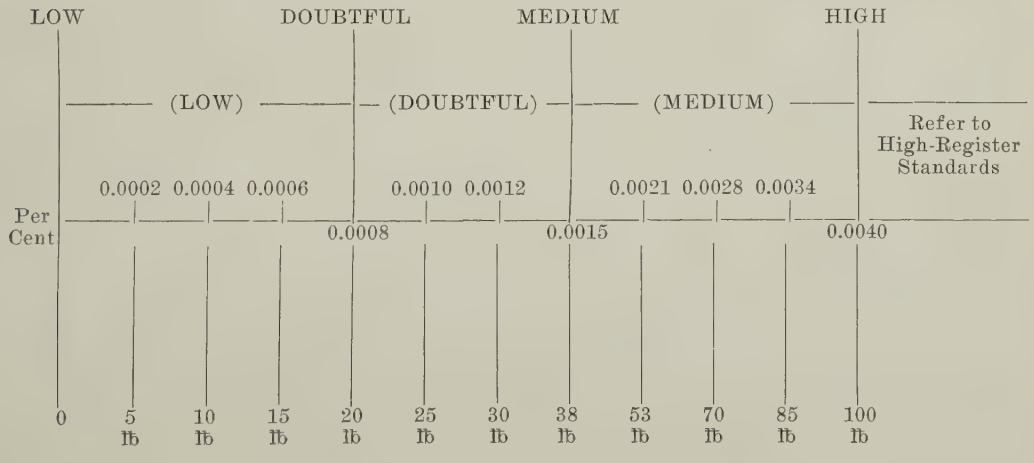
1. Store stannous chloride solution, Reagent 4, P_2O_5, in a dark place.
2. Color standards should be kept in closed boxes when not in actual use.
3. When the small pieces of tin in the bottles of stannous chloride solution disappear, reject the solution and obtain fresh supplies. Order this reagent in small quantities.

TABLE FOR HIGH-REGISTER SOIL P_2O_5 STANDARDS

Standard No.	I		II		III		IV		V		VI		VII	
P_2O_5 Values	Per Cent	lb/ a-ft.	Per Cent	lb/ a-ft.	Per Cent	lb/ a-ft.	Per Cent	lb/ a-ft.	Per Cent	lb/ a-ft.	Per Cent	lb/ a-ft.	Per Cent	lb/ a-ft.
	.004	100	.006	150	.008	200	.010	250	.012	300	.014	350	.016	400
AFTER DILUTION														
One volume of developed solution plus one volume of Reagent 4, P_2O_5.	.016	400	020	500	.024	600	.028	700	.032	800				

Color intensity greater than VII should be recorded as "greater than .032 per cent, 800 pounds, per acre-foot," or (>.032 per cent, >800 pounds per acre-foot).

INTERPOLATED PERCENTAGE FIGURES FOR P_2O_5 (R. C. M.) AND THEIR EQUIVALENTS IN POUNDS OF P_2O_5 PER ACRE-FOOT



Phosphate Fixation

Equipment Required

1 Support, iron, 6 in. x 9 in.
1 Dropping bottle, pipette stopper with nipple, 30-ml. cap.
12 Prescription ovals, 2 oz., etched.
12 Vials, shell, tall form.
1 Set phosphate fixation color standards, in box (include pc. of black sateen).
1 Metal soil cup, 5-gram cap.
2 pkg. Munktell No. 3, 11-cm. filter paper.
1 Burette, dispensing, 250-ml. cap.

1 Burette cover, for 250-ml. dispensing burette.
1 Pipette, Exax, volumetric, 1-ml. cap.
1 Vial block.
1 Spatula, stainless steel, 4-inch blade.
1 Funnel support.
1 gal. Series 100 fixation solution.
1 gal. Series 500 fixation solution.
1 gal. Series 1000 fixation solution.
1 pt. Ammonium molybdate solution.
½ pt. Stannous chloride solution in g.s.b.
1 Clamp, burette, castaloy, large, with rubber-covered jaws.
12 Funnels, 65 mm.

Additional Equipment Recommended

2 Burettes, dispensing, 250-ml. cap. (with proper supports).
2 Covers, for 250-ml. dispensing burettes.

Equipment Required for Phosphate Fixation Not Included in Potash or Phosphoric Acid Ensembles

12 Prescription ovals, 2 oz., etched.
1 Set phosphate fixation color standards, in box (include pc. of black sateen).
1 Pipette, Exax, volumetric, 1-ml. cap.
1 gal. Series 100 fixation solution.
1 gal. Series 500 fixation solution.
1 gal. Series 1000 fixation solution.
1 pt. Ammonium molybdate solution.

Preliminary

Three series of phosphate fixation solutions are provided which vary in concentration with respect to phosphoric acid. Since each of these solutions is applied in the same volume to the same quantity of soil it represents varying applications of phosphate fertilizer to the field, as follows:

Series 100 — 1,500 lb P_2O_5 per acre-foot
Series 500 — 7,500 lb P_2O_5 per acre-foot
Series 1000 — 15,000 lb P_2O_5 per acre-foot

I. Series 100: When unfamiliar with the general range of phosphate fixing power of the soils to be studied, or whenever general experience has shown that the soils of a region frequently yield index values less than 90 with Series 100 solution, it is desirable to employ this solution. In cases where Series 100 solution almost invariably gives index values of 90, it is not necessary to start with this fixation solution.

II. Series 500: Whenever the Series 100 index value is 60 or higher, make additional determinations with Series 500 solution. If the Series 500 index is below 20, use Series 100 solution if that has not already been done.

III. Series 1000: Whenever the Series 500 index value is 40 or higher, determinations should be made with the Series 1000 solution.

Procedure

1. Transfer 30 ml. of the proper phosphate solutions from a 250-ml. dispensing burette to a labeled bottle.
2. Fill a 5-gram metal cup with prepared soil and level off with a stainless steel spatula.
3. Transfer the soil to the bottle containing phosphate solution, and proceed:
   (a) With Series 100: Shake 1 minute and let stand overnight. Shake 1 minute again before filtering.
   (b) With Series 500 and 1000: Shake 3 minutes; let stand for 24 hours; shake 3 minutes again; let stand another 24 hours and finally (after a total duration of 48 hours) shake 3 minutes before filtering.
4. Filter through No. 3 Munktell filter paper, 11 cm., into a clean shell vial, tall form. Discard approximately the first 5 ml. and then collect filtrate in the tube until it is about an inch from the top of the tube.
5. Place the vials in a numbered vial block and add 1 ml. ammonium molybdate solution, mix and add 1 drop of stannous chloride solution. Mix well; let stand ½ minute, or until maximum color is developed.
6. Place on phosphate illuminator and compare with phosphate fixation color standards.
7. Record index value *with series number* of phosphate solution, e.g., if index number is 20 when Series 100 is used, the recorded item becomes: 100-20.

Precautions

1. Store stannous chloride solution in a dark place.
2. Color standards should be kept in closed boxes when not in actual use.
3. When the small piece of tin in the bottle of stannous chloride solution disappears, reject the solution and obtain fresh supplies. Order this reagent in small quantities.

Rapid Estimation of Phosphate in Cane Juice

Equipment Required

12 Funnels, short stem, 90 mm.
1 Funnel support.
12 Beakers, Pyrex, 50-ml. cap.
24 Vials, shell, tall form.
1 Burette, dispensing, 250-ml. cap.
1 Cover for 250-ml. dispensing burette.
1 Support, iron, 6 in. x 9 in.
1 Clamp, burette, castaloy, large, with rubber-covered jaws.
1 Pipette, special, 0.5-ml. cap.
1 Box No. 12, Whatman folded filter paper, 18.5 cm.
1 Bottle, dropping, pipette stopper with nipple, 30-ml. cap.

1 Set phosphate-in-juice color standards (with box).
1 Phosphate illuminator.
2 Pipettes, volumetric, Exax, 5-ml. cap.
1 gal. Reagent 4, P_2O_5.
½ pt. Stannous chloride solution in g. s. b.
1 qt. Distilled water.
¼ lb. Cane juice preservative.

Equipment Required for Phosphate-in-Cane-Juice Estimations Not Included in Soil Phosphate or Potash Ensembles

12 Funnels, short stem, 90 mm.
1 Pipette, transfer, 0.5 ml. cap.
1 Box No. 12 Whatman folded filter paper, 18.5 cm.
1 Set phosphate in juice color standards (in box).
2 Pipettes, volumetric, Exax, 5-ml. cap.
1 qt. Distilled water.
¼ lb Cane juice preservative.

Procedure

Cane juice should either be fresh or preserved with proper preservative, supplied for the rapid chemical methods.

1. Filter cane juice through Whatman No. 12, 18.5-cm. folded filter paper.
2. With the pipette, deliver into a phosphate vial an 0.5-ml. aliquot of filtrate.
3. Then, from the dispensing burette, add Reagent 4, P_2O_5, to a volume of about 5 mm. from the top.
4. Shake well and add 1 drop of stannous chloride solution.
5. Shake again and immediately compare with the phosphate color standards for cane juice, using the P_2O_5 illuminator. Note result. Then add another drop of stannous chloride solution, shake and make comparison again.
6. If the color is too dark for comparison, use a smaller aliquot until a comparable color is obtained.
7. Refer to the table and by means of the aliquot taken and its P_2O_5 equivalent represented by the color standard, obtain the per cent P_2O_5 by volume in the juice, recording the result indicated by the maximum color developed.
8. If the juice contains more than .064 per cent P_2O_5, mix 5 ml. of the filtered juice with an equal volume of distilled water, using a 5-ml. pipette. Proceed with steps Nos. 2 to 6. Refer to the table and multiply the value by 2 to obtain the percentage of P_2O_5.
9. If the value obtained by following step No. 8 is in excess of .128 per cent P_2O_5, mix 5 ml. of the filtered juice with 10 ml. of distilled water and proceed. Multiply the value in the table by 3.

Precautions

1. Store stannous chloride solution and Reagent 4, P_2O_5, in a dark place.
2. Color standards should be kept in closed boxes when not in actual use.

3. When the small pieces of tin in the bottles of stannous chloride solution disappear, reject the solution and obtain fresh supplies. Order this reagent in small quantities.

PERCENTAGE P_2O_5 BY VOLUME IN CANE JUICE

Stand No.	Ml. of Cane Juice Taken				
	0.5	0.4	0.3	0.2	0.1
1	0.006	0.008	0.011	0.016	0.032
2	0.010	0.012	0.016	0.024	0.048
3	0.013	0.016	0.021	0.032	0.064
4	0.016	0.020	0.027	0.040	0.080
5	0.019	0.024	0.032	0.048	0.096
6	0.022	0.028	0.037	0.056	0.112
7	0.026	0.032	0.043	0.064	0.128
8	0.029	0.036	0.048	0.072	0.144

Rapid Estimation of Phosphoric Acid in Boiler Water

1 Filter the collected specimen twice through Whatman No. 12, folded filter paper.

2. Transfer 2 ml. of the cold filtrate into a tall form, phosphate vial.

3. Make up the volume to about ¼ inch from the top by adding reagent No. 40.

4. Mix the contents of vial thoroughly by inverting several times, stoppering the vial with the index finger.

5. Add 1 drop of stannous chloride solution.

6. Mix well and immediately compare with the phosphate color standards for cane juice, using the phosphate illuminator. Note result.

7. Add another drop of stannous chloride solution, shake and make comparison again.

8. Consult the table and by referring to the number of the standard which matches the maximum color developed, obtain the concentration of phosphoric acid in the sample tested.

TABLE FOR PHOSPHORIC ACID IN BOILER WATER

Standard No.	P.P.M. P_2O_5
1	16
2	24
3	32
4	40
5	48
6	56
7	64
8	72

Rapid Estimation of Phosphoric Acid in Filter Cake

Note: The equipment listed below is already part of R.C.M. assemblies for determining P_2O_5 in soil and in crusher juice and nitrogen in soil.

Equipment Required

1 Only 5-gram metal soil cup.
6 Only 125-ml. Erlenmeyer flasks.
6 Only 65-mm. glass funnels.
1 Only filtering rack.
1 Only 250-ml. dispensing burette for N/2 HCl solution.
1 Only 250-ml. dispensing burette for Reagent 4, P_2O_5.
1 Only iron support with 2 large clamps for 250-ml. dispensing burettes.
6 Only 100-ml. Pyrex beakers.
1 Only 0.5-ml. special juice pipette.
1 doz. Phosphate vials.
1 box Whatman No. 12, 15-cm. filter paper.
1 gal. N/2 hydrochloric acid solution.
1 qt. Reagent 4, P_2O_5.
¼ pt. Stannous chloride solution.
1 Only 30-ml. dropping bottle, pipette stopper with nipple, for stannous chloride solution.
1 Set phosphate-in-cane-juice color standards.
1 Phosphate illuminator.
1 Block for holding phosphate vials.
1 Only 10-ml. Exax volumetric pipette.

Procedure

1. Obtain a representative sample of the filter cake.

2. Break up large lumps by crumbling with hand. Mix material thoroughly. The sample is now ready for analysis without drying.

3. Fill a 5-gram metal soil cup with filter cake. Pack cup by pressing slightly the material with blade of spatula. Scrape off excess specimen to level of cup. Transfer specimen into a 125-ml. Erlenmeyer flask.

4. Add 50 ml. N/2 hydrochloric acid solution. Shake 1 minute and let stand 1 hour.

5. At the end of 1 hour, shake for 1 minute and filter through Whatman No. 12, 15-cm. filter paper, collecting the filtrate in a 100-ml. Pyrex beaker. (Same type of glassware and filter paper as used for available N in soil determination.) Mix the filtrate by stirring.

6. With a special 0.5-ml. juice pipette, deliver in each of 2 phosphate vials, 0.2-ml. aliquots of filtrate.

7. From a dispensing burette, add Reagent 4, P_2O_5, to a volume of about ½ inch from the top of the vial. Mix well by inverting tubes several times.

8. Develop color by adding 1 drop of stannous chloride reagent to each vial. Mix well and immediately compare with P_2O_5-in-cane-juice color standards, using the illuminator.

9. If the color is too dark or too light for comparison, use a smaller or larger aliquot until the shade developed is within range of the standards.

10. Refer to Table A and with reference to the aliquot taken, obtain the pounds P_2O_5 per cubic yard of filter cake.

11. When the phosphate content is above that indicated in Table A, further analysis may be made by diluting a portion of the original filtrate.

12. To proceed with further analysis: Pipette 10 ml. of the filtrate into a 125-ml. Erlenmeyer flask. Add 40 ml. N/2 hydrochloric acid solution, mix solution thoroughly. Use diluted solution for analysis as in steps Nos. 6 to 9, inclusive. Refer to Table B and with reference to the aliquot of diluted solution taken, obtain the pounds P_2O_5 per cubic yard of filter cake.

POUNDS P_2O_5 PER CUBIC YARD OF FILTER CAKE

TABLE A

(Using Original Filtrate)

	ml. of Original Filtrate Used For Analysis				
Std. No.	0.5	0.4	0.3	0.2	0.1
1	1.0	1.3	1.7	2.7	5.4
2	1.7	2.0	2.7	4.0	8.1
3	2.0	2.7	3.4	5.4	10.8
4	2.7	3.4	4.4	6.7	13.5
5	3.0	4.0	5.4	8.1	16.2
6	3.7	4.7	6.1	9.4	18.9
7	4.4	5.4	7.1	10.8	21.6
8	4.7	6.1	8.1	12.1	24.3

TABLE B

(Using Diluted Filtrate)

	ml. of Diluted Filtrate Used For Analysis				
Std. No.	0.5	0.4	0.3	0.2	0.1
1	5.4	6.7	9.0	13.5	27.0
2	8.1	10.1	13.5	20.2	40.4
3	10.8	13.5	18.0	27.0	53.9
4	13.5	16.8	22.5	33.7	67.4
5	16.2	20.2	27.0	40.4	80.9
6	18.9	23.6	31.5	47.2	94.4
7	21.6	27.0	35.9	53.9	107.8
8	24.3	30.3	40.4	60.7	121.3

Rapid Estimation of Potash in Soils

Equipment Required

1 Metal soil cup, 2.5-gram cap.
1 Metal soil cup, 5.0-gram cap.
1 Metal soil cup, 10-gram cap.
1 Burette, dispensing, 250-ml. cap.
1 Cover for 250-ml. dispensing burette.
24 Flasks, Erlenmeyer, 125-ml. cap.
24 Beakers, Pyrex, 50-ml. cap.
24 Funnels, short stem, 65 mm.
2 pkg. Munktell No. 3, 9-cm. filter paper.

2 pkg. Munktell No. 3, 11-cm. filter paper.
24 Vials, shell, short form.
1 Pipette, medicine dropper, 1-ml. cap. (calib.).
1 Burette, Exax, 50-ml. cap.
1 Cover for 50-ml. burette.
4 Inclined blocks.
1 Potash rotator, electric, Model "G."
1 Potash Illuminator.
2 Supports, iron, 6 in. x 9 in.
1 Clamp, burette, Lincoln.
1 Clamp, burette, castaloy, large, with rubber-covered jaws.
1 Funnel support.
1 Spatula, stainless steel, 4-inch blade.
1 Dropping bottle, amber, pipette stopper with nob at tip, 30-ml. cap.
1 gal. Reagent 1, K_2O.
¼ pt. Reagent 2, K_2O g. s. b.
1 pt. Reagent 3, K_2O.

Procedure

1. Fill a 2.5-gram metal cup with prepared soil and level off with a stainless steel spatula.

2. Transfer the soil to a 125-ml. Erlenmeyer flask.

3. Add 10 ml. of Reagent 1, K_2O, from a 250-ml. dispensing burette.

4. Swirl for ½ minute.

5. Filter through Munktell No. 3 filter paper, 9 cm., into a 50-ml. beaker.

6. With the calibrated medicine dropper, transfer 1-ml. portions of the filtrate to the bottom of each of 2 vials, short form.

7. Add 4 drops of Reagent 2, K_2O, to each of the vials and mix thoroughly.

8. Immediately place the vials on an inclined block. Hold the block horizontally, thus keeping the vials inclined at the proper angle. Touch the inner side of each vial with the tip of a 50-ml. burette and allow 1 ml. of Reagent 3, K_2O, to flow down the side so that very little mixing takes place. The addition of Reagent 3, K_2O, should require approximately 4½ to 5 seconds with the rate of flow as uniform as possible. Two distinct liquid layers should be visible. In case the layers are broken or a cloudiness appears between the 2 layers, discontinue and repeat steps Nos. 6 to 8 until 2 distinct liquid layers are obtained.

9. Place the vials in slots Nos. 1 and 3 of the potash rotator.

10. If a second soil extract has been prepared, it may be tested at the same time, using slots Nos. 2 and 4.

11. Start the instrument and stop it exactly 30 seconds later.

12. Allow the vials to stand for about 30 seconds and then transfer to the illuminator.

13. Move the slide back and forth over the lined chart and at the same time sight down through the columns of test liquid.

If heaviest lines cannot be seen, reading is 4.

If heaviest lines can be seen, but medium lines are invisible, reading is 3.

If medium lines can be seen but light lines are invisible, reading is 2.

If light green lines can be seen, reading is 1.

14. If the 2 readings do not agree, repeat the duplicate determinations until 2 or more identical readings are obtained.

15. If 2 or more readings of 2 or 3 are obtained, estimate the K_2O content by reference to the attached table and average the values obtained.

16. If a reading of 1 is obtained, prepare fresh extracts using successively larger amounts of soil with sufficient quantities of Reagent 1, K_2O, to obtain 5 ml. or more of filtrate. The desirable amounts to use are indicated in the table. Proceed with steps Nos. 6 to 14 until readings of 2 or 3 are obtained. If a reading of 1 is still obtained using a ratio of 1:1 (20 gm. soil to 20 ml. Reagent 1, K_2O), record the potash content as "Low," $<.003$ per cent and <75 pounds K_2O per acre-foot.

17. If a reading of 4 is obtained, prepare fresh extracts of 2.5 gm. of soil to 15, 20, 25, 30, 35 and 40 ml. of Reagent 1, K_2O, until readings of 2 or 3 are obtained. If a reading of 4 is still obtained with 2.5 grams of soil to 40 ml. of Reagent 1, K_2O, the potash content can be recorded as "High," $>.056$ per cent, >1400 pounds per acre-foot.

Precaution

Store Reagent 2, K_2O, in a dark place. Order in small quantities.

APPROXIMATE PERCENTAGES OF K_2O AND THEIR CORRESPONDING VALUES IN POUNDS OF K_2O PER ACRE-FOOT

Ratio Soil : Reagent	Readings	Per Cent K_2O	Lbs. K_2O per acre-foot	Approx. Group
20 gm. 20 ml.	1	<.003	< 75	
	2	.003	75	
	3	.004	100	
	4	>.004	>100	
15 gm. 20 ml.	1	<.004	<100	
	2	.004	100	—LOW
	3	.005	125	
	4	>.005	>125	
10 gm. 20 ml.	1	<.006	<150	
	2	.006	150	
	3	.007	175	
	4	>.007	>175	
7.5 gm. 20 ml.	1	<.009	<225	
	2	.009	225	
	3	.010	250	—DOUBTFUL
	4	>.010	>250	
2.5 gm. 10 ml.	1	<.012	<300	
	2	.012	300	
	3	.014	350	
	4	>.014	>350	
2.5 gm. 15 ml.	1	<0.18	<450	
	2	.018	450	
	3	.021	525	—MEDIUM
	4	>.021	>525	
2.5 gm. 20 ml.	1	<.025	<625	
	2	.025	625	
	3	.028	700	
	4	>.028	>700	
2.5 gm. 25 ml.	1	<.031	<775	
	2	.031	775	
	3	.035	875	
	4	>.035	>875	
2.5 gm. 30 ml.	1	<.037	<925	
	2	.037	925	
	3	.042	1050	—HIGH
	4	>.042	>1050	
2.5 gm. 35 ml.	1	<.044	<1100	
	2	.044	1100	
	3	.049	1225	
	4	>.049	>1225	
2.5 gm. 40 ml.	1	<.050	<1250	
	2	.050	1250	
	3	.056	1400	
	4	>.056	>1400	

Rapid Estimation of Potash in Cane Juice

Equipment Required

12 Funnels, short stem, 90 mm.
1 Funnel support.
1 box Whatman No. 12, 18.5-cm. folded filter paper.
1 Burette, dispensing, 250-ml.
1 Cover for 250-ml. dispensing burette.
12 Beakers, Pyrex, 50-ml. cap.
1 Pipette, transfer, straight type, 1-ml. cap.
2 Pipettes, medicine dropper, calibrated to 1 ml.
24 Vials, shell, short form.
2 Burette, Exax, 50 ml.
2 Covers for 50-ml. Exax burette.
1 Potash rotator, electric, Model "G."
1 Potash illuminator.
1 Clamp, burette, castaloy, large, with rubber-covered jaws.
1 Bottle, dropping, pipette stopper with nipple, for Reagent 2, K_2O.
2 Supports, iron, 6 in. x 9 in.
1 Clamp, burette, Lincoln.
1 gal. Reagent 10, K_2O.
¼ pt. Reagent 2, K_2O g.s.b.
1 pt. Reagent 3, K_2O.
¼ lb Cane juice preservative.

Additional Equipment Required for Potash in Cane Juice Assembly Not Included in Potash and Phosphate in Soil Ensembles

12 Funnels, short stem, 90 mm.
1 box Whatman No. 12, 18.5-cm. folded filter paper.
1 Pipette, transfer, straight type, 1-ml. cap.
1 gal. Reagent 10, K_2O.

Procedure

1. Use fresh, untreated juice or juice preserved with the proper preservative supplied for rapid chemical methods.

2. Obtain a representative sample. Shake well.

3. Pour into a dry filter paper (Whatman No. 12, 18.5 cm. folded) on a 90-mm. funnel and collect in a clean, dry 50-ml. beaker.

4. Transfer 1 drop of juice to the bottom of a clean potash vial and 2 drops to a second vial, using a medicine dropper held vertically.

5. With a calibrated medicine dropper add 1 ml. Reagent 10, K_2O.

6. Add 4 drops of Reagent 2, K_2O, to each of the vials and mix, using a few quick shakes.

7. Immediately place the vials on an inclined block. Hold the block horizontally, thus keeping the vials inclined at the proper angle. Touch the inner side of each vial with the tip of a 50-ml. burette and allow 1 ml. of Reagent 3, K_2O, to flow down the side so that very little mixing takes place. The addition of Reagent 3,

K_2O, should require approximately 5 to 6 seconds with the rate of flow as uniform as possible. Two distinct liquid layers should be visible. In case the layers are broken or a cloudiness appears between the 2 layers, discontinue and repeat steps Nos. 4 to 7 until 2 distinct liquid layers are obtained.

8. Place the vials in slots Nos. 1 and 3 of the potash rotator.

9. If a second sample of juice has been filtered, it may be tested at the same time, using slots Nos. 2 and 4.

10. Start the instrument and stop it exactly 30 seconds later.

11. Allow the vials to stand for about 30 seconds and then transfer to the illuminator.

12. Move the slide back and forth over the lined chart and at the same time sight down through the columns of test liquid.

If heaviest lines cannot be seen, reading is 4.

If heaviest lines can be seen, but medium lines are invisible, reading is 3.

If medium lines can be seen, but light lines are invisible, reading is 2.

If light green lines can be seen, reading is 1.

13. Record the readings. These are preliminary tests to indicate the correct dilutions to use in the following section of the procedure.

14. Transfer 1-ml. portions of the juice into clean 50-ml. beakers and add the volume of Reagent 10, K_2O, indicated by the table below:

TABLE OF DILUTIONS

1-Drop Vial		2-Drop Vial	
Reading	ml. Reagent 10, K_2O	Reading	ml. Reagent 10, K_2O
2	20	1	10, or less
3	25	2	15
4	30, or greater	3	15

15. Mix well and transfer 1-ml. portions of the diluted juice into each of 2 potash vials, using a calibrated medicine dropper.

16. Repeat steps Nos. 6 to 12 and record the readings.

17. If the 2 readings do not agree, repeat the duplicate determinations until 2 or more identical readings are obtained.

18. If 2 or more readings of 2 or 3 are obtained, estimate the potash content by reference to the table "Potash in Cane Juices" and average the values obtained.

19. If readings of 1 are obtained, add a smaller volume of Reagent 10, K_2O, with a second 1-ml. portion of juice, as suggested in the table "Potash in Cane Juices."

20. If readings of 4 are obtained, add a larger volume of Reagent 10, K_2O, with 1 ml. of the juice.

21. Repeat until 2 or more readings of 2 or 3 are obtained. Refer to the table and average the percentage values indicated.

22. If readings of 4 are obtained with 1 dilution and readings of 1 with the next higher dilution, average the results indicated by the corresponding 2 and 3 readings. For example:

We obtain readings of 1 with 20 ml. of reagent and readings of 4 with 15 ml. of reagent. Average the values .16 per cent and .14 per cent, yielding the result, .15 per cent.

23. If, with no dilution, readings of 1 are obtained, record the result as <.01 per cent K_2O. If, with an addition of 50 ml. reagent 10, K_2O, readings of 4 are obtained, record the result as >.43 per cent K_2O.

Precaution

Store Reagent 2, K_2O, in a dark place. Order in small quantities.

TABLE FOR POTASH IN CANE JUICES

ml. Reagent 10, K_2O	Per Cent Potash (K_2O)			
	Reading 1	Reading 2	Reading 3	Reading 4
0	<.01	.01	.01	>.01
3	<.03	.03	.03	>.03
5	<.05	.05	.05	>.05
7	<.06	.06	.07	>.07
10	<.08	.08	.09	>.09
12	<.10	.10	.11	>.11
15	<.12	.12	.14	>.14
20	<.16	.16	.18	>.18
25	<.20	.20	.22	>.22
30	<.23	.23	.26	>.26
35	<.27	.27	.31	>.31
40	<.31	.31	.35	>.35
50	<.38	.38	.43	>.43

Rapid Estimation of Potash in Molasses

(Sample to be weighed out on an analytical balance.)

Equipment Required

Items required for potash-in-juice determinations:

1 flask, volumetric, 100-ml. cap.

Procedure

1. Weigh out 5.0 grams of molasses in a weighing dish or small tared beaker.
2. Add about 25 ml. distilled water and mix well by stirring.
3. Transfer to a 100-ml. volumetric flask, washing in with distilled water. Make volume up to the mark, stopper the flask and mix.
4. Proceed according to the directions for the rapid estimation of potash in cane juices, steps Nos. 3 to 17.

Note: Potash may be determined on molasses using the solution prepared for the rapid estimation of total nitrogen in molasses, step No. 3, *after samples for the nitrogen determinations have been secured.* But do not determine total nitrogen on a filtered sample.

5. Proceed with steps Nos. 18 to 22, referring to the table below and substituting for the example in step No. 22, the following:

We obtain readings of 1 with 20 ml. of reagent and readings of 4 with 15 ml. of reagent. Average the values 3.2 per cent and 2.8 per cent, yielding the result,

3.0 per cent, or the values 64 and 56, yielding the result 60 pounds K_2O per ton of molasses.

TABLE FOR POTASH IN MOLASSES

ml. Reagent 10, K_2O	Reading of 2		Reading of 3	
	Per cent	lb/ton	Per cent	lb/ton
10	1.6	32	1.8	36
12	2.0	40	2.2	44
15	2.4	48	2.8	56
20	3.2	64	3.6	72
25	4.0	80	4.4	88
30	4.6	92	5.2	104
35	5.4	108	6.2	124
40	6.2	124	7.0	140
50	7.6	152	8.6	172

Rapid Estimation of Potash in Irrigation Water

Equipment Required

1 Only 6-inch electric hot plate.
1 Only 250-ml. graduated cylinder.
6 Only 400-ml. Pyrex beakers.
6 Only 600-ml. Pyrex beakers.
3 Only rubber policemen.
6 Only 40-mm. glass funnels.
1 Only filtering rack (10 funnels).
1 Only 1-ml. pipette, graduated to 1/10 ml.
1 pkg. Munktell No. 3, 7-cm. filter paper.
1 pkg. Munktell No. 3, 9-cm. filter paper.
1 Only 2-ml. Mohr pipette.
1 doz. Potash vials.
1 Only potash rotator.
1 Only potash illuminator.
1 gal. Reagent 1, K_2O solvent.
¼ pt. Reagent 2, K_2O g.s.b.
1 pt. Reagent 3, K_2O.

Additional Equipment Required for Potash in Irrigation Water Assembly Not Included in Other Rapid Method Ensembles

1 Only 250-ml. graduated cylinder.
6 Only 400-ml. Pyrex beakers.
6 Only 600-ml. Pyrex beakers.
3 Only rubber policemen.
1 Only 1-ml. pipette, graduated to 1/10 ml.
1 pkg. Munktell No. 3, 7-cm. filter paper.

Note to Analysts:

You will observe that this rapid method for determination of potash in irriga-

tion waters is essentially a modification of the standard procedure for estimating potash in soil in which the potash rotator, potash illuminator, and other standard equipment are employed.

Step No. 4 reads: "Add 5, 10 or 15 ml. Reagent 1, K_2O." To explain: If the volume of residue remaining is bulky after step No. 2 in the process, or if previous knowledge of the water indicates that the potash content may be high, then the amount of Reagent 1, K_2O, may be taken as 15 ml. instead of 5 or 10 ml. If, on the other hand, the bulk of precipitate is small or potash content is believed to be reasonably low, then use 5 ml. or 10 ml. of the reagent and proceed as directed. For instance: Were you to be working with a pump water from central Maui, where dry residue is high and potash is also high, you would probably decide to use 15 ml. of Reagent 1, K_2O. If, on the other hand, you were dealing with Waiahole Ditch water from the island of Oahu, where potash is rather low in concentration and where other salts are equally low (hence small residue on drying), you would find it expedient to employ 5 ml. of Reagent 1, K_2O, in bringing the potash from the dry residue into solution.

Step No. 5 reads: "Rub sides and bottom of the beaker well, using rubber policeman." The purpose of this step is to insure intimate contact of the residue in the beaker with Reagent 1, K_2O, the latter being an excellent solvent for potash. In many cases you will find that a portion of the residue will not go into solution. This is to be expected and we believe will not occasion any serious difficulty regarding the accuracy of the test, for Reagent 1, K_2O, will very effectively dissolve the potash in this test, providing you bring it in intimate contact with the reagent for a few seconds.

In analyzing a mountain water or other irrigation supply which may be very low in potash, a larger volume will have to be taken for evaporation. In some waters we have found it necessary to use as much as 750 ml. in order to obtain enough for making an accurate analysis.

A word of explanation regarding the data sheet accompanying the method of analysis: You will find the data sheet divided into 3 sections, one of which is to be used when 5 ml. of Reagent 1, K_2O, are employed in Step No. 4, another when 10 ml. of Reagent 1, K_2O, have been used and the third where 15 ml. of this reagent were required to effect solution of the potash. In each case you will find notations at the top of the data sheet, heading columns of data, in which any one of several amounts of filtrate may have been used, varying from 1.0 ml. in decreasing increments of 0.1 ml. down to and including 0.1 ml. Under these headings you will find data already calculated for readings of "2" and "3" corresponding to potash present in parts per million and pounds per million gallons. A reading in this case (as in the case of the soil and juice potash analyses) represents the designations you give the tests when making your turbidity determination with the potash illuminator. You will note we have omitted readings "1" and "4" from the data sheet. This has been done because of the fact that were you to obtain either one of these values it would necessitate a repetition of the analysis simply because the values lie at the extremities of the lined chart and as a consequence cannot be counted upon always as being absolutely correct. A reading of "1," for instance, may be exactly that value, but in all probability it is much less than "1." Likewise, a reading of "4" may be just "4" in relation to the remainder of the chart, but most probably it is higher than "4." Conversely, a reading of "2" or "3" places the determination in an intermediate

position between the extremes of "1" and "4" and hence may be accepted as reliable. To illustrate the discussion in this paragraph, let us take an hypothetical case: A sample of water is evaporated to dryness and the residue brought into solution with 10 ml. Reagent 1, K_2O; 0.6 ml. of the filtrate was used in the test and a reading of "2" was made on the illuminator. You would report, then, that the water contained 5.3 parts per million or 44 pounds of potash as K_2O per million gallons.

When an amount greater than 250 ml. of irrigation water is used for evaporation preparatory to making the analysis, a factor will have to be employed for reducing the readings on the data sheet. For instance, if 500 ml. water are taken, divide the result obtained by 2; if 750 ml. are used, by 3, etc.

Rapid Method for the Determination of Potash in Pump Waters

1. Measure out 250 ml. of the water into a 400-ml. beaker.*
2. Place on hot plate and carefully evaporate to dryness.
3. Cool.
4. Add 5, 10 or 15 ml. Reagent 1, K_2O.
5. Rub sides and bottom of the beaker well, using rubber policeman.
6. Filter through Munktell No. 3 paper into a 50-ml. beaker. (For 5 ml. Reagent 1, K_2O, use 7-cm. filter paper. For 10 and 15 ml. Reagent 1, K_2O, use 9-cm. filter paper.)
7. Transfer 0.5 ml. of the filtrate into a potash vial.
8. Make up to 1 ml. with Reagent 1, K_2O. Shake.
9. Add 4 drops Reagent 2, K_2O. Shake 3 times.
10. Add 1 ml. Reagent 3, K_2O, down the inclined side of the vial.
11. Put vial into the potash rotator and run for ½ minute.
12. The readings are made identically as for other potash kits, using the potash illuminator.
13. Under the columns for the number of ml. of Reagent 1, K_2O, added to the residue and the fractional part of a ml. of the filtrate taken, read directly the K_2O content in parts per million (p.p.m.), or pounds per million gallons.
14. If the reading is 4, take 0.4, 0.3, 0.2 or 0.1 ml. of the filtrate and make up to 1 ml. in each case, using Reagent 1, K_2O, until 3 and 2 readings are obtained.
15. For "1" reading, use 0.6, 0.7, 0.8, 0.9 or 1.0 ml. portions as above until 2 and 3 readings are obtained.

* For mountain water, use about 500 ml. or more and proceed as usual.

RAPID DETERMINATION OF POTASH IN PUMP WATER

(250 ml. water evaporated to dryness.)

5 ml. Reagent 1, K_2O, added to residue and filtered

Reading	K_2O content	ml. of filtrate taken									
		1.0 ml.	0.9 ml.	0.8 ml.	0.7 ml.	0.6 ml.	0.5 ml.	0.4 ml.	0.3 ml.	0.2 ml.	0.1 ml.
	p.p.m.	1.6	1.8	2.0	2.3	2.7	3.2	4.0	5.3	8.0	16.0
2	lb/mil. gal.	13	15	17	19	23	27	33	44	67	133
	p.p.m.	1.8	2.0	2.3	2.6	3.0	3.6	4.5	6.0	9.0	18.0
3	lb/mil. gal.	15	17	19	22	25	30	38	50	75	150
	10 ml. Reagent 1, K_2O, added to residue and filtered										
	p.p.m.	3.2	3.6	4.0	4.6	5.3	6.4	8.0	10.7	16.0	32.0
2	lb/mil. gal.	27	30	33	38	44	53	66	89	133	267
	p.p.m.	3.6	4.0	4.5	5.1	6.0	7.2	9.0	12.0	18.0	36.0
3	lb/mil. gal.	30	33	38	43	50	60	75	100	150	300
	15 ml. Reagent 1, K_2O, added to residue and filtered										
	p.p.m.	4.8	5.3	6.0	6.9	8.0	9.6	12.0	16.0	24.0	48.0
2	lb/mil. gal.	40	44	50	58	67	80	100	133	200	400
	p.p.m.	5.4	6.0	6.8	7.7	9.0	11.0	13.5	18.0	27.0	54.0
3	lb/mil. gal.	45	50	57	64	75	91	112	150	225	450

NOTE: In all cases reading "1" is less than the respective "2" reading, and reading "4" is greater than the respective "3" reading.

Rapid Estimation of Potash and Phosphoric Acid in Mill Ash

Equipment Required

6 Only 100-ml. Pyrex beakers.
1 Analytical balance.*
6 Only 125-ml. Erlenmeyer flasks.
6 Only 65-mm. glass funnels.
1 Only 250-ml. dispensing burette.
1 Only 10-ml. pipette.
1 pkg. Munktell No. 3, 12.5-cm. filter paper.*
3 Only medicine droppers calibrated to 1 ml.
10 Only short-type, shell vials, 2-dram cap.
6 Only 50-ml. Pyrex beakers.
1 Only 50-ml. burette.
1 Only 1-ml. pipette graduated to 0.01 ml.
1 Only 2-ml. Mohr pipette.
1 Only potash rotator.
1 Only potash illuminator.
10 Only tall-type, shell vials marked to hold 8 ml. solution.*
6 Only 50-ml. volumetric flasks.*
6 Only rubber stoppers for volumetric flasks.*
1 Only 0.5-ml. pipette, graduated into 0.5-, 0.4-, 0.3-, 0.2- and 0.1 ml. divisions.
1 Only filtering rack.
1 Only phosphate vial rack.
1 Only phosphate illuminator.
1 set phosphate-in-cane-juice color standards.
1 Data sheet.*
1 gal. Reagent 1, K_2O solvent.
¼ pt. Reagent 2, K_2O g. s. b.
1 pt. Reagent 3, K_2O.
1 gal. Reagent 4, P_2O_5.
1 gal. N/2 hydrochloric acid.
1 gal. distilled water.
1 pt. Stannous chloride, g. s. b.

* Note: Not included in other rapid chemical methods assemblies.

Preparation of Sample for Analysis

A. Obtain representative ash sample (about 250 grams—½ lb.)
B. Break up sample into powder form as much as possible.
C. Spread sample out evenly on a clean piece of paper.
D. Divide sample into 4 equal parts.
E. Take opposite quarters and mix well.
F. Repeat steps D and E twice. (The portion to be used for analysis will be equivalent to 1/16 of the original sample.)
G. Analyze resulting specimen for K_2O and P_2O_5 by Methods I and II, respectively.

I. Determination of Potash in Mill Ash

1. Weigh 1 gram of sample from (G) and place in a 125-ml. flask.
2. Add 50 ml. of Reagent 1, K_2O solvent.
3. Shake 1 minute.
4. Filter into a clean, dry beaker through Munktell No. 3, 12.5-cm. filter paper.
5. Transfer 0.5 ml. of the filtrate to the bottom of a short type, comparison vial.
6. Make up to a full ml., using Reagent 1, K_2O solvent.
7. Add 4 drops of Reagent 2, K_2O, to one of the vials and mix thoroughly.
8. Immediately incline vial with contents and let 1 ml. of Reagent 3, K_2O, flow down the side so that very little mixing takes place. (Two distinct liquid layers indicate correct technic.)
9. Place the vial in a slot of the potash agitator.
10. Start the instrument. (If a spring-wound rotator is used, it is important to rewind the spring for every 2 to 4 determinations.)
11. Run instrument for 30 seconds, the timing to start when the rotating cylinder makes permanent contact with the surrounding wooden guide-piece.
12. At the end of 30 seconds, stop the rotator.
13. Turn on the light of the potash illuminator.
14. Remove the vial from the rotator and insert into a slot of the illuminator, then move back and forth over the lined chart and at the same time sight down through the column of the test liquid.

If heaviest lines cannot be seen, reading is 4.

If heaviest lines can be seen but medium lines are invisible, reading is 3.

If medium lines can be seen but light green lines are invisible, reading is 2.

If light green lines can be seen, reading is 1.

Important: The solution in the vial must be smooth. If the solution is curdy, or a crystalline precipitate forms, repeat, using less filtrate.

15. If a reading of 1 is obtained, use 0.6, 0.7, 0.8, 0.9 or 1.0 ml. of the filtrate, making up to a full ml. in each case with Reagent 1, K_2O solvent.
16. If a reading of 4 is obtained, use 0.4, 0.3, 0.2 or 0.1 ml. of the filtrate, making up to a full ml. in each case with Reagent 1, K_2O solvent.
17. Add Reagents 2 and 3, K_2O, as before, mix in the rotator and take reading in the potash illuminator.
18. Refer to Table 1 for per cent potash (K_2O).
19. In case a 4 reading is obtained when 0.2 or 0.1 ml. of the filtrate is used, pipette out 10 ml. of the filtrate and add 40 ml. of Reagent 1, K_2O solvent, to make up to a total volume of 50 ml. Mix thoroughly.
20. Take 1.0, 0.9, 0.8 ml., etc., of this solution in a comparison vial, make up to a full ml. with Reagent 1, K_2O solvent, and continue the test as before.
21. Use Table 2 for potash content of ash.

TABLE FOR RAPID ESTIMATION OF POTASH IN MILL ASH
(1 gram ash, 50 ml. Reagent 1, K_2O solvent.)

TABLE 1

Reading	K_2O content	ml. of filtrate taken										
		1.0 ml.	0.9 ml.	0.8 ml.	0.7 ml.	0.6 ml.	0.5 ml.	0.4 ml.	0.3 ml.	0.25 ml.	0.2 ml.	0.1 ml.
2	Per Cent	.38	.42	.47	.54	.63	.75	.94	1.25	1.5	1.9	3.8
3	Per Cent	.43	.47	.53	.61	.71	.85	1.1	1.4	1.7	2.1	4.3

NOTE: Reading 1 is less than the respective 2 readings.
Reading 4 is greater than the respective 3 readings.

TABLE 2
(1 gram ash, 50 ml. Reagent 1, K_2O solvent, 10 ml. filtrate plus 40 ml. Reagent 1 K_2O solvent.)

Reading	K_2O content	ml. of filtrate taken										
		1.0 ml.	0.9 ml.	0.8 ml.	0.7 ml.	0.6 ml.	0.5 ml.	0.4 ml.	0.3 ml.	0.25 ml.	0.2 ml.	0.1 ml.
2	Per Cent	1.9	2.1	2.3	2.7	3.1	3.8	4.7	6.3	7.5	9.4	19.0
3	Per Cent	2.1	2.4	2.7	3.0	3.5	4.3	5.3	7.1	8.5	10.6	21.0

NOTE: Reading 1 is less than the respective 2 readings.
Reading 4 is greater than the respective 3 readings.

II. Determination of Phosphoric Acid, P_2O_5 in Mill Ash

1. Weigh 1 gram of prepared sample from (G) and transfer to a 125-ml. flask.
2. Add 50 ml. N/2 HCl to the sample in the flask.
3. Stir for 3 minutes and filter through a Munktell No. 3, 12.5-cm. filter paper into a 100-ml. beaker.
4. Transfer 10 ml. of the filtrate by means of a 10-ml. pipette into a 50-ml. volumetric flask and make up to its 50-ml. mark with distilled water.
5. Mix the solution in the flask several times.
6. By means of a special 0.5-ml. pipette, transfer a 0.3-ml. portion of the diluted solution (5) to a special phosphate vial.
7. Then add Reagent 4, P_2O_5, into the vial up to the mark indicating an 8-ml. volume.
8. Mix well by inverting tube several times.
9. Add 1 or 2 drops of stannous chloride and mix again.
10. Immediately compare the developed color with the same set of color standards employed for the rapid chemical analysis of phosphate in cane juice.

Note A: In case the color developed is darker than the No. 8 standard tube, take the next smaller portion of filtrate, 0.2 ml., and repeat steps Nos. 9 to 12.

Note B: Conversely, when the color is too light, use a larger portion of 0.4 ml. or 0.5 ml. and repeat steps Nos. 9 to 12.

11. Refer the number on the standard matched and the number of ml. taken to the data sheet for mill ash analysis and obtain percentages of P_2O_5 in the sample.

TABLE FOR RAPID ESTIMATION OF PHOSPHORIC ACID IN MILL ASH

(Figures represent percentages of P_2O_5 in Ash)

P_2O_5 Standard Tube Matched	ml. of Solution Taken for Comparison				
	0.1 ml.	0.2 ml.	0.3 ml.	0.4 ml.	0.5 ml.
1	..	4.0	2.7	2.0	1.6
2	..	6.0	4.0	3.0	2.4
3	..	8.0	5.3	4.0	3.2
4	..	10.0	6.7	5.0	4.0
5	..	12.0	8.0	6.0	4.8
6	..	14.0	9.3	7.0	5.6
7	..	16.0	10.7	8.0	6.4
8	..	18.0	12.0	9.0	7.2

Rapid Estimation of Calcium in Soils

Equipment Required

12 Flasks, Erlenmeyer, 125-ml. cap.
1 pkg. No. 3 Munktell filter paper, 11 cm.
12 Funnels, 65 mm.
1 Funnel support.
24 Vials, shell, short form.
12 Beakers, Pyrex, 50-ml. cap.
1 Pipette, Mohr, 2-ml. cap., marked to deliver in 0.25-ml. portions.

1 Pipette, medicine dropper, calibrated to 1 ml.
1 Spatula, stainless steel, 4-inch blade.
1 Calcium illuminator.
1 Standard calcium chart.
1 Burette, dispensing, 250-ml. cap.
1 Cover for 250-ml. dispensing burette.
1 Burette, Exax, 50-ml. cap.
1 Cover for 50-ml. burette.
2 Supports, iron, 6 in. x 9 in.
1 Burette clamp, castaloy, large, with rubber-covered jaws.
1 Burette clamp, Lincoln.
1 Metal soil cup, 2.5-gm. cap.
1 gal. Reagent 13, Ca.
1 pt. Reagent 14, Ca.

Additional Equipment Recommended

1 Metal soil cup, 5.0-gm. cap.
1 Metal soil cup, 10.0-gm. cap.

Equipment Required for Calcium Soil Determinations Not Included in Potash or Phosphate in Soil Ensembles

1 Pipette, Mohr, 2-ml. cap.
1 Calcium illuminator.
1 Standard calcium chart.
1 gal. Reagent 13, Ca.
1 pt. Reagent 14, Ca.

Procedure

1. Fill a 2.5-gram metal cup with prepared soil and level off with a stainless steel spatula. Transfer to a 125-ml. Erlenmeyer flask.
2. Add 30 ml. of Reagent 13, Ca, from a 250-ml. dispensing burette.
3. Shake with a rotating motion for ½ minute, giving approximately 100 swirls.
4. Immediately filter through Munktell No. 3, 11-cm. filter paper into a 50-ml. beaker.
5. Transfer 1.0 ml. into a comparison tube by means of a 2-ml. Mohr pipette.
6. Add 1.0 ml. of Reagent 13, Ca, from a 50-ml. burette.
7. Add 1.0 ml. of Reagent 14, Ca, from a calibrated medicine dropper.
8. Immediately close the tube with your thumb and shake the tube with sufficient rapidity to allow 30 shakes in less than 7 seconds.
9. Let stand for ½ minute.
10. With the light turned on, place the tube in the middle slot of the illuminator slide. Close the other 2 slots with your fingers.
11. Move back and forth over the lined chart and at the same time sight down through the column of the test liquid:

If heavy lines cannot be seen, reading is 4.

If heavy lines can be seen, but medium lines are invisible, reading is 3.

If medium lines can be seen, but light lines are invisible, reading is 2.

If light lines can be seen, reading is 1.

12. Whatever the initial reading, it is necessary to check or amplify the result by repeating steps Nos. 5 to 11 with different aliquots of the filtrate until for each soil sample consecutive readings of both 2 and 3 are obtained. The appropriate lines must be clearly seen before being recorded as 2 or 3. With any aliquot make up the volume to 2 ml. with Reagent 13, Ca, before adding 1 ml. of Reagent 14, Ca. In using a burette to add Reagent 13, Ca, run out enough of the reagent into a flask provided for the purpose so as to start from a full ml. mark.

13. If an initial reading of 4 is obtained, use aliquots of 0.75 ml., 0.50 ml., and 0.25 ml. until readings of 3 and 2 are obtained.

14. If the initial reading is 3, use the aliquots above (step No. 13) until a reading of 2 is obtained.

15. In case of an initial reading of 1, use aliquots of 1.25 ml., 1.50 ml., 1.75 ml., and 2.0 ml. until readings of 2 and 3 are obtained.

16. If the initial reading is 2, use the aliquots above (step No. 15) until a reading of 3 is obtained.

17. If necessary, prepare a fresh extract of 5 grams, 10 grams, 20 grams or 30 grams of soil with 30 ml. of Reagent 13, Ca, and proceed as above until consecutive readings of both 2 and 3 are obtained.

18. With the recorded readings of 2 and 3, refer to the table and estimate the percentage and pounds per acre-foot of CaO by taking the lowest figure represented.

Examples:

(A) 2.5-gram extraction:

Aliquots	Readings	Per cent CaO	Final Result	
			% CaO	lb CaO/a-ft.
1.00	2	0.22	0.22	5500
1.25	3	0.25	(lowest value)	

(B) If a reading of 2 is finally obtained for a 2-ml. aliquot, prepare another extraction with 5 grams of soil to 30 ml. Reagent 13, Ca.

2.5-gram extraction:

Aliquots	Readings	Per cent CaO
1.00	1	<0.22
1.25	1	<0.17
1.50	1	<0.14
1.75	1	<0.12
2.00	2	0.11

5.0-gram extraction:

Aliquots	Readings	Per cent CaO	Final Result	
			% CaO	lb CaO/a-ft.
1.00	2	0.11		
1.25	2	0.087	0.087	2175
1.50	3	0.10	(lowest value)	

TABLE FOR RAPID ESTIMATION OF CALCIUM IN SOILS

(Results as per cent and lb CaO/a-ft.)

Extract 30 ml. No.13,Ca	Rdg.	0.25 ml.		0.50 ml.		0.75 ml.		1.00 ml.		1.25 ml.		1.50 ml.		1.75 ml.		2.00 ml.	
		%	lb	%	lb	%	lb	%	lb	%	lb	%	lb	%	lb	%	lb
	4	>1.25	>31250	>0.62	>15500	>0.42	>10500	>0.31	>7750	>0.25	>6250	>0.21	>5250	>0.18	>4500	>0.16	>4000
2.5 grams	3	1.25	31250	0.62	15500	0.42	10500	0.31	7750	0.25	6250	0.21	5250	0.18	4500	0.16	4000
(1 cup)	2	0.87	21750	0.43	10750	0.29	7250	0.22	5500	0.17	4250	0.14	3500	0.12	3000	0.11	2750
	1	<0.87	<21750	<0.43	<10750	<0.29	< 7250	<0.22	<5500	<0.17	<4250	<0.14	<3500	<0.12	<3000	<0.11	<2750
	4																
5 grams	3							>0.16	>4000	>0.13	>3250	>0.10	>2500	>0.089	>2225	>0.078	>1950
(2 cups)	2							0.16	4000	0.13	3250	0.10	2500	0.089	2225	0.078	1950
	1							0.11	2750	0.087	2175	0.072	1800	0.062	1550	0.054	1350
								<0.11	<2750	<0.087	<2175	<0.072	<1800	<0.062	<1550	<0.054	<1350
	4																
10 grams	3							>0.078	>1950	>0.062	>1550	>0.052	>1300	>0.044	>1100	>0.039	> 975
(4 cups)	2							0.078	1950	0.062	1550	0.052	1300	0.044	1100	0.039	975
	1							0.054	1350	0.043	1075	0.036	900	0.031	775	0.027	675
								<0.054	<1350	<0.043	<1075	<0.036	< 900	<0.031	< 775	<0.027	< 675
	4																
20 grams	3									>0.031	> 775	>0.026	> 650	>0.022	> 550	>0.020	> 500
(8 cups)	2									0.031	775	0.026	650	0.022	550	0.020	500
	1									0.022	550	0.018	450	0.015	375	0.014	350
										<0.022	< 550	<0.018	< 450	<0.015	< 375	<0.014	< 350
	4																
30 grams	3											>0.017	> 425	>0.015	> 375	>0.013	> 325
(12 cups)	2											0.017	425	0.015	375	0.013	325
	1											0.012	300	0.010	250	0.009	225
												<0.012	< 300	<0.010	< 250	<0.009	< 225

Rapid Estimation of Calcium in Filter Cake

(Sample to be weighed out on an analytical balance)

Equipment Required

2 Pipettes, volumetric, Exax, 5-ml. cap.
6 Beakers, 50-ml. cap.
1 Burette, Exax, 50-ml. cap.
1 Cover for 50-ml. burette.
1 Bottle, dropping, 60 ml., with medicine dropper calibrated to 1-ml. cap.
12 Vials, shell, short form.
1 Calcium illuminator, and chart.
1 Pipette, Mohr, 2-ml. cap., marked to deliver in 0.25-ml. portions.
6 Flasks, Erlenmeyer, 125-ml. cap.
6 Funnels, 65 mm.
1 Funnel support.
1 Burette, dispensing, 250-ml. cap.
1 Cover for 250-ml. dispensing burette.
6 Beakers, Pyrex, 100-ml. cap.
1 Box Whatman No. 12, 15-cm. folded filter paper.
1 gal. N/2 hydrochloric acid solution.
1 qt. Reagent 13, Ca.
1 qt. Reagent 19, Ca.
½ pt. Reagent 14, Ca.

Procedure

1. Refer to the method for the rapid estimation of phosphoric acid in filter cake (sample to be weighed out on an analytical balance). Prepare an extract according to steps Nos. 1 to 6 of this procedure. (Both phosphoric acid and calcium can be determined by using portions of this extract.)

2. Pipette 5 ml. of the extract into a 50-ml. beaker. Add 5 ml. of Reagent 19, Ca, with another 5-ml. pipette. Mix thoroughly.

3. With a special 2-ml. Mohr pipette, transfer successively increasing aliquots to short-form vials. Add from a 50-ml. burette sufficient of Reagent 13, Ca, to bring the total volume in each vial to 2 ml. Four vials should be prepared for each determination.

4. Add 1 ml. Reagent 14, Ca, from a calibrated medicine dropper to one of the vials. Immediately close the tube with the thumb and shake the tube with sufficient rapidity to allow 30 shakes in approximately 7 seconds. Repeat this procedure with each of the other tubes in turn.

5. Place the illuminator slide so that the slots open on the No. 3 lines of the calcium chart. Place the first 3 vials in the slots, and make observations. Remove the vial which shows the lines plainly and place the remaining vial in its place.

6. Note the aliquot which produces a turbidity such that the No. 3 lines are barely, but yet definitely visible (in other words, a high No. 3 reading is to be obtained). Note the next increasing aliquot in which the lines are not visible, a No. 4 reading.

7. Refer to the table and obtain the results for the 2 readings. These results are expressed as per cent CaO (lbs. CaO per 100 lbs. filter cake and as pounds CaO per ton of filter cake). The final value is the average of the 2 readings.

TABLE FOR CALCIUM IN FILTER CAKE

Aliquot	Reading of 3 Calcium, CaO		Reading of 4 Calcium, CaO	
	Per cent	lb/ton	Per cent	lb/ton
1.50	.24	4.8	.35	7.0
1.40	.26	5.2	.37	7.4
1.30	.28	5.6	.40	8.0
1.20	.30	6.0	.43	8.6
1.10	.33	6.6	.48	9.6
1.00	.36	7.2	.52	10.4
.90	.40	8.0	.58	11.6
.80	.45	9.0	.65	13.0
.70	.51	10.2	.74	14.8
.60	.60	12.0	.87	17.4
.50	.72	14.4	1.04	20.8
.45	.80	16.0	1.16	23.2
.40	.90	18.0	1.30	26.0
.35	1.03	20.6	1.49	29.8
.30	1.20	24.0	1.73	34.6
.25	1.44	28.8	2.08	41.6
.20	1.80	36.0	2.60	52.0
.15	2.40	48.0	3.47	69.4
.10	3.60	72.0	5.20	104.0

Estimation of Soil Reaction (pH)

Equipment Required

1 Soil reaction color chart.
2 Porcelain spot plates.
3 Porcelain test plates, triple channel.
6 Glass rods with fine points.
1 Spoon, horn, small.
1 Spatula, stainless steel, 4-inch blade.
1 Brush, camel hair, pencil, medium size.
1 pkg. Munktell No. 3, 9-cm. filter paper.
4 Bottles, dropping, with pipettes, 30-ml. cap.
½ pt. Bromthymol blue indicator.
½ pt. Bromcresol green indicator.
½ pt. Phenol red indicator.
½ pt. Chlorphenol red indicator.
½ pt. Distilled water.

Equipment Required for Estimation of Soil Reaction Included in Soil Potash or Phosphate Assemblies

1 Spatula, stainless steel, 4-inch blade.
1 pkg. Munktell No. 3, 9-cm. filter paper.

Preliminary Test, Using the Spot Plate

1. Using a small spoon or a stainless steel spatula, place prepared soil in a depression of the spot plate until it is about ⅓ full.

2. Add bromthymol blue indicator drop by drop until the soil is saturated; add 3 drops more.

3. Pick the plate up and shake it for about 10 seconds (12 counts) by a motion which may be described as a combined rocking and rotary motion.

4. Put the plate back on the table and using the large end of the glass rod transfer a drop of the liquid to the flat surface adjoining the depression.

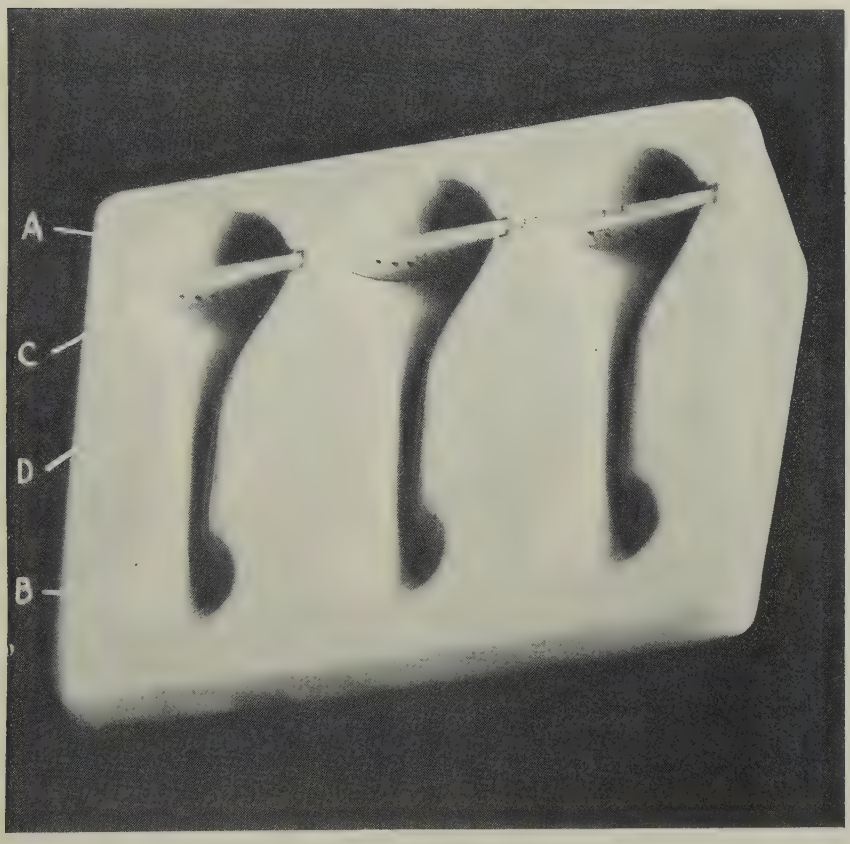
5. Compare the color of the drop with the colors of the bromthymol blue color chart.

6. If a match is not obtained, repeat steps Nos. 1 to 6, using chlorphenol red or phenol red, depending on whether the pH of the soil is below or above the bromthymol blue range, respectively. The test can be further extended on the acid side by using the bromcresol green indicator. If the pH appears to be below 3.8 or above 8.2, bromphenol blue or thymol blue indicators and their respective color charts may be used. These are available and will be supplied upon request.

7. When a match is obtained with any indicator near the mid-portions of the corresponding color chart, proceed with the triple channel block for the final and accurate determinations.

8. If a match is obtained but near the end portions of the chart, the test should be repeated with the indicator next in the series to determine which of the two will give readings closer to the middle portions of the chart before proceeding with the use of the triple-channel block.

Procedure for Use of Triple-Channel Block



A—Depression
B—Reservoir
C—Partition
D—Channel

1. By means of a small horn spoon or stainless steel spatula, fill with soil the 3 depressions marked "A" on the drawing. Press the soil lightly against the partition "C" with the spoon or spatula to make certain that the soil is in complete contact with the partition.

2. Using a camel hair brush, remove completely any soil that may have fallen into channel "D" or reservoir "B."

3. Add the indicator, predetermined by the preliminary test, drop by drop until the soil is saturated; add 2 or 3 additional drops.

4. Apply the sharp end of the glass rod very lightly against the minute openings of the partition "C" on the side opposite the soil, and gently draw the solution out into channel "D." With the large end of the glass rod gently draw a small portion of the solution into reservoir "B."

Precaution: Draw the liquid into the channel and reservoir; *do not* rub the partition or the surface of the porcelain block with the glass rod.

5. Immediately compare the color of the liquid in depression "B" with the color chart of the respective indicator used.

Note: In case the color of the liquid in reservoir "B" appears deeper in intensity than those of the chart, transfer a drop of the solution by means of the glass rod to the flat surface adjacent to the reservoir and compare the color of this drop with the colors of the chart. When the liquid comes out too pale in color, as often happens, with the bromthymol blue indicator, wipe off the liquid with a sheet of clean filter paper and draw out more solution, applying more indicator to the soil if necessary.

6. When a match is obtained near the mid-portions of the chart (for example: 6.4 to 7.2 on the bromthymol blue chart) record the reading. Determinations should at least be made in triplicate, two of which should check before reporting the result.

7. If a match is obtained, but near the end portions of a chart, make a few additional determinations using the indicator next in the series. When the results obtained by the two indicators do not agree, report the average of the two results as the pH of the soil.

8. Interpretation of results: A reaction of pH 7.0 indicates a neutral soil, that is, one which is neither acid nor alkaline. Values below pH 7.0 denote acidity, the intensity of acidity increasing as the numbers decrease. Values above 7.0 denote alkalinity, the intensity of alkalinity increasing as the numbers increase.

Precautions

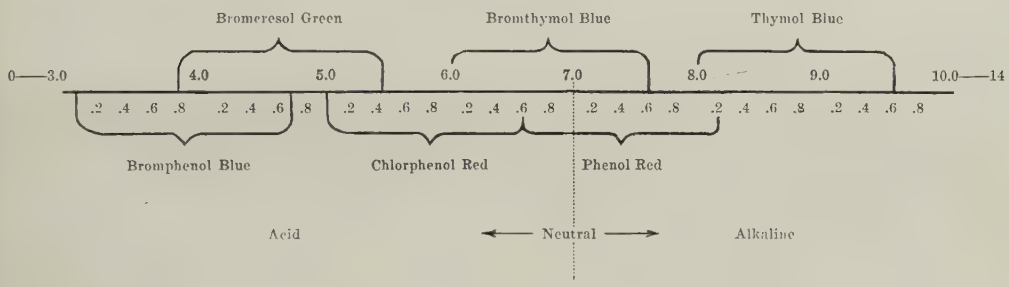
1. The color chart should be kept in the envelope provided for the purpose when not in use since exposure to light accelerates fading of the colors.

2. Make readings in a bright, natural, diffused daylight. Readings made in dazzling sunlight or in artificial light may be incorrect.

3. Do not conduct the test while ammonia or acid fumes are present in the laboratory.

SOIL REACTION (pH)

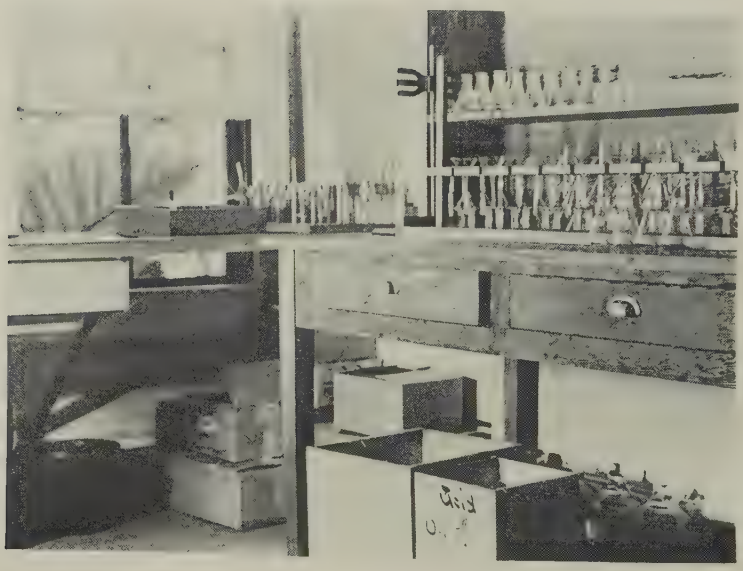
Indicator Series:



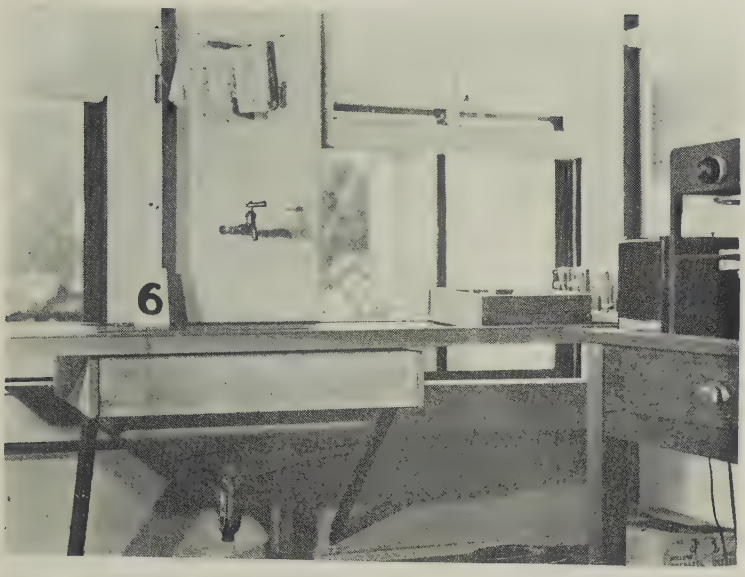
PLANTATION R. C. M. LABORATORIES

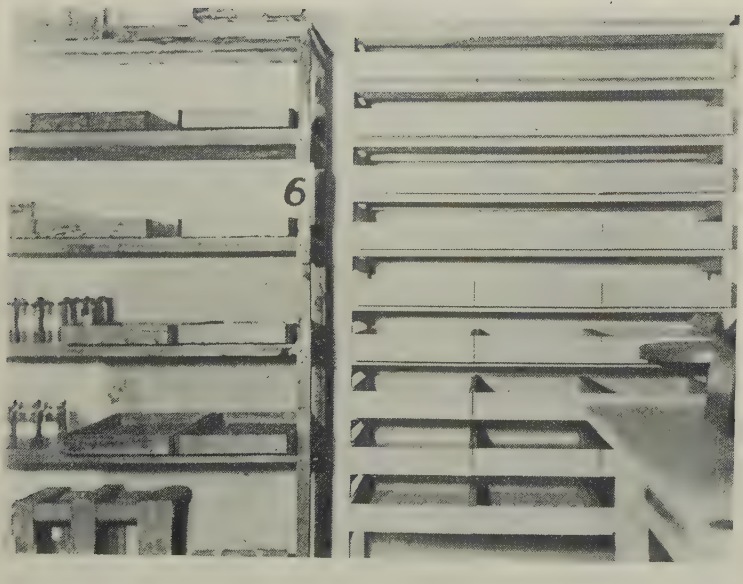
The following illustrations depict views of some of the plantation R. C. M. laboratories.

A view of the R.C.M. laboratory at Pioneer Mill Company, Ltd.

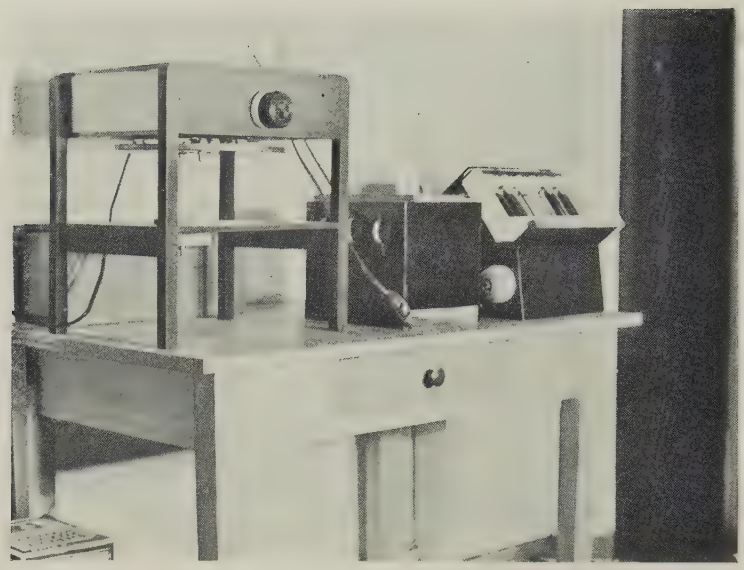


Views of R.C.M. laboratory of island representative, Experiment Station, H.S.P.A. (Maui).

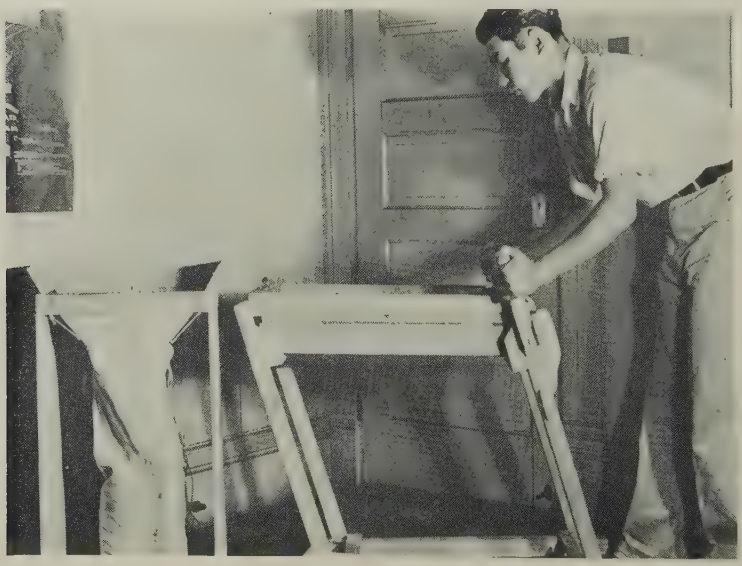




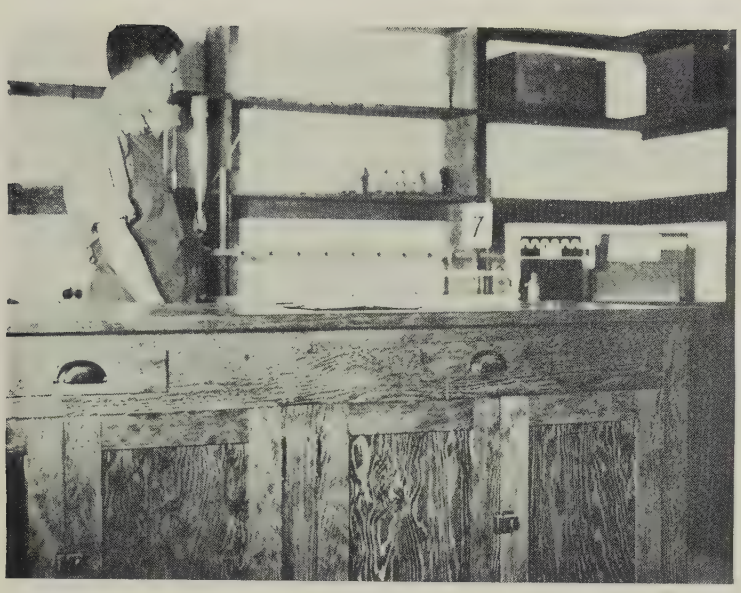
Laboratory views, island representative, Experiment Station, H.S.P.A. (Maui).



R.C.M. soil equipment. (Left to right, Potash Rotator, Potash Illuminator, Phosphate Illuminator.)



Preparing soils for analysis.

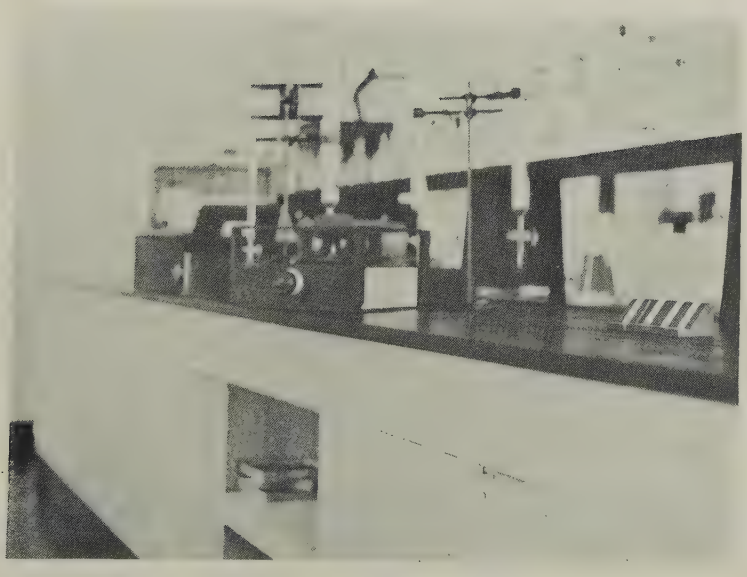


A view of the Waiakea Mill Company R.C.M. laboratory.

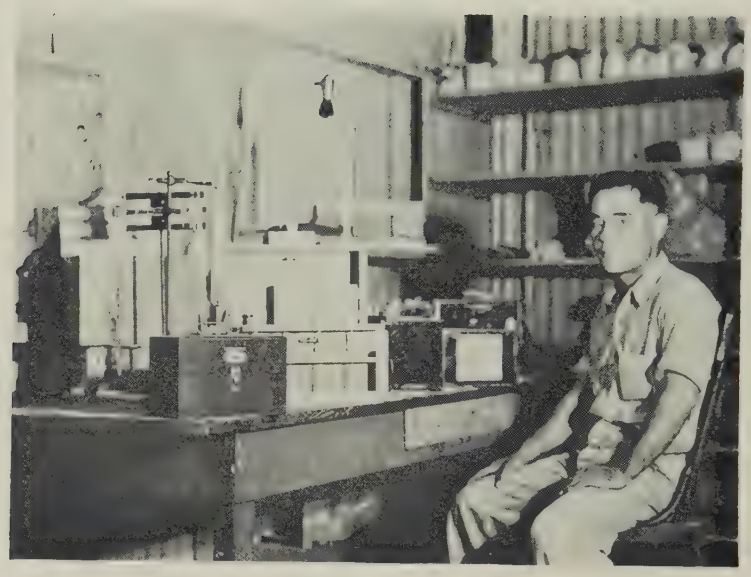
A corner of Olaa Sugar Company, Ltd., R.C.M. laboratory.

One end of the R.C.M. laboratory at Hutchinson Sugar Plantation Company.

An outside view of the Hutchinson Sugar Plantation Company R.C.M. laboratory.

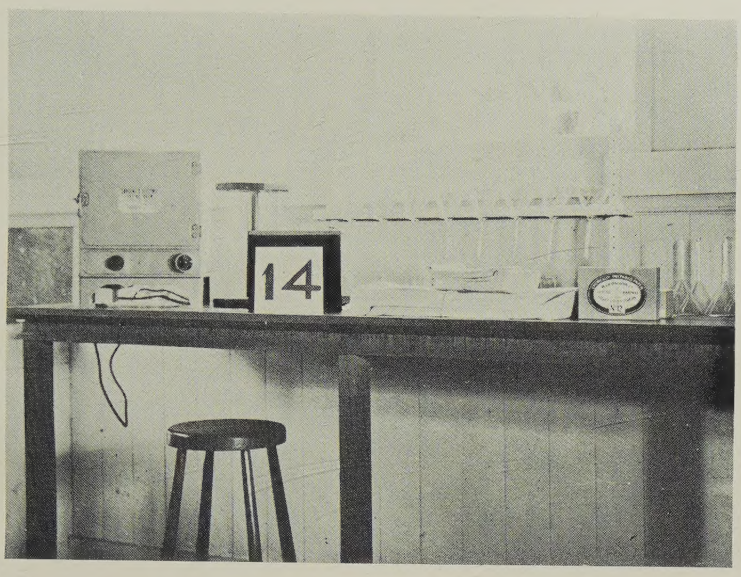


Two views of the R.C.M. laboratory at Hawaiian Agricultural Company.

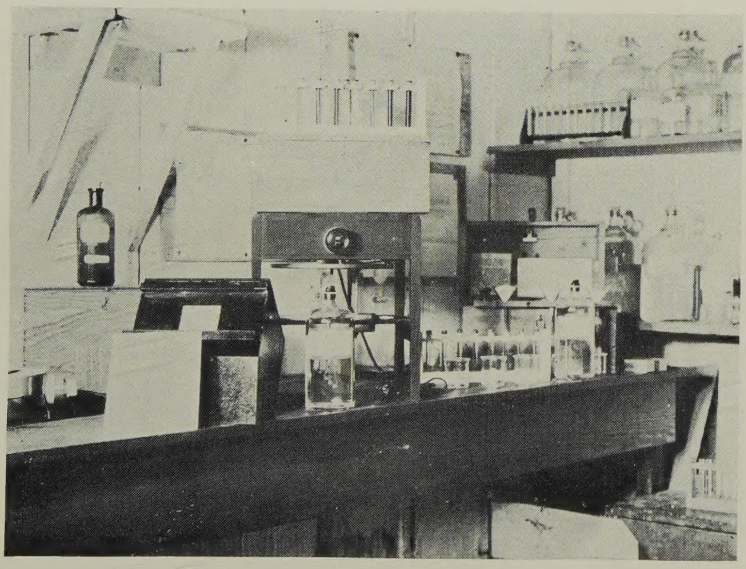


View of the R.C.M. laboratory at Kilauea Sugar Plantation Company.

A corner of the R.C.M. laboratory at Paauhau Sugar Plantation Company.

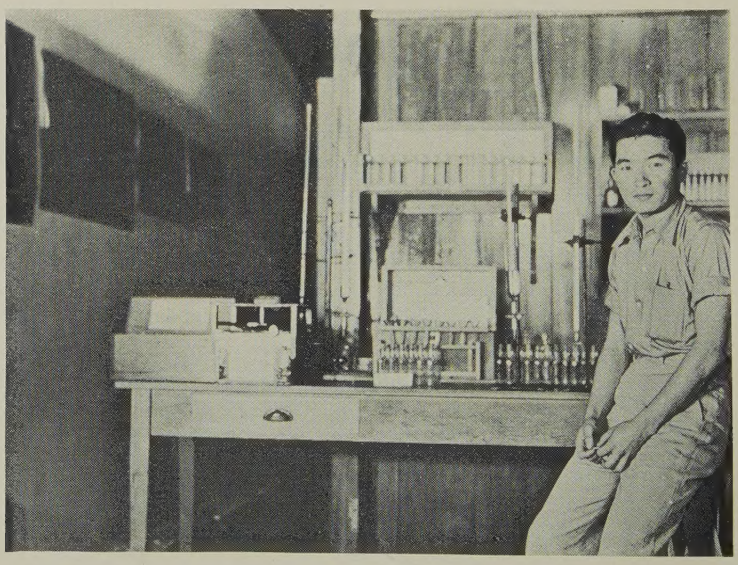


A portion of the R.C.M. laboratory of the island representative, H.S.P.A. (Hilo).

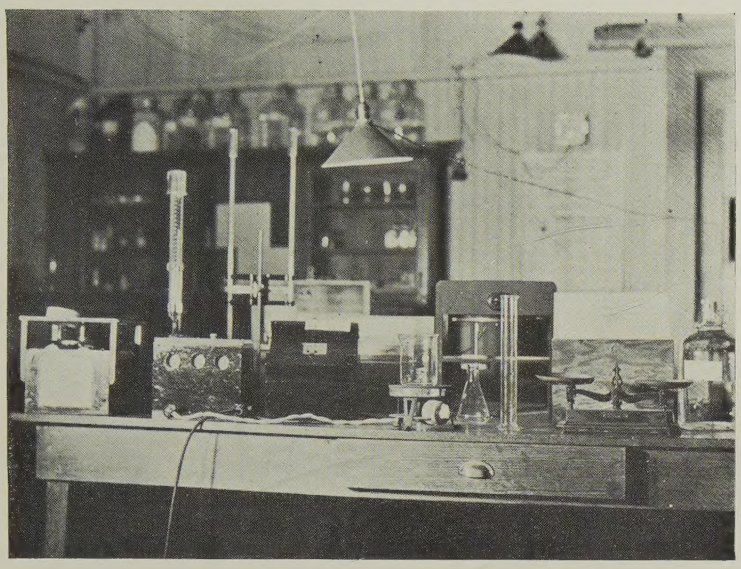


A view of the R.C.M. laboratory at Hakalau Plantation Company.

A corner of the R.C.M. laboratory at Honomu Sugar Company.



One end of the R.C.M. laboratory at The Lihue Plantation Company, Ltd.



The R.C.M. laboratory of the island representative of Experiment Station, H.S.P.A. (Kauai).

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(2) ————————, 1932. A test for replaceable and water-soluble potassium in soils. Jour. Amer. Soc. Agr., 24: 312-316.

(3) Hance, F. E., 1933. Phosphate fixation in Hawaiian soils. The Hawaiian Planters' Record, 37: 182-196.

(4) ————————, 1936. The Third International Congress of Soil Science. The Hawaiian Planters' Record, 40: 127-136.

(5) Handbook of Hawaiian Soils. 1935. Association of Hawaiian Sugar Technologists, pp. 92-96.

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Sugar Prices

96° CENTRIFUGALS FOR THE PERIOD
MARCH 23, 1936, TO JUNE 3, 1936.

Date	Per Pound	Per Ton	Remarks
March 23, 1936	3.60¢	$72.00	Puerto Ricos.
" 30	3.75	75.00	Puerto Ricos.
May 4	3.72	74.40	Puerto Ricos.
June 3	3.76	75 20	Cubas.